Journal

American Leather Chemists Association

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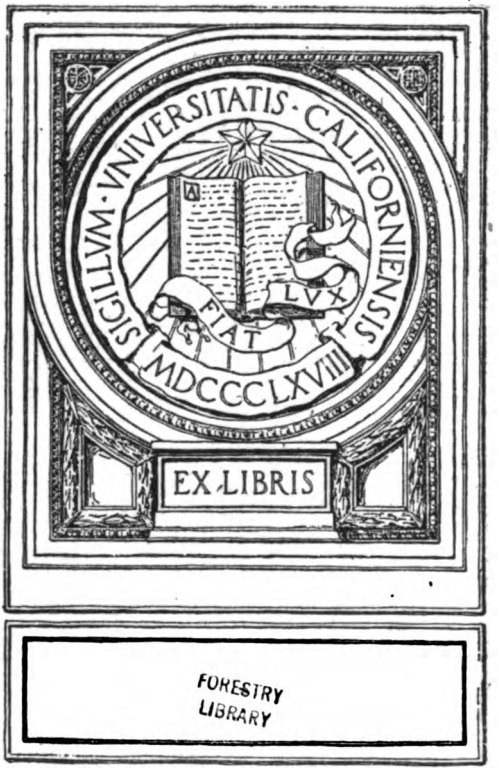

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Association

VOLUME I, 1906

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THE JOURNAL OF THE AMERICAN LEATHER CHEMISTS' ASSOCIATION

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Journal of the

American Leather Chemists Association

Vol. I. JANUARY, 1906 No. 1

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EDITORIAL

The committee appointed at the Washington Convention to inquire into the advisability of issuing an Association Journal, having reported favorably on the project, the Council authorized a monthly publication to be issued under the direction of the staff as above composed, and this issue of the JOURNAL marks the beginning of the realization of the hopes of the Council.

To fully realize their hopes the editors must have the coöperation of all our members. We expect papers from all the members, and must have them, if the JOURNAL is to have individuality, standing, and interest, and is to be other than a mere record of

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Association proceedings, or a review of our contemporaries. While we cannot expect to begin with an entirely original publication, yet we hope to make the original articles the feature of the JOURNAL, and the digests of analogous contemporary publications will be corollaries to the original proposition.

There is certainly a field for our publication in this country, and if the JOURNAL can cover the field and supply the demand for a magazine of its nature, we shall expect tangible evidence of its appreciation. If the Editors do not purvey the quality or quantity of subject matter demanded in this field, they will appreciate honest criticism, and trust that the character of the JOURNAL will profit thereby.

As Editors, we are beginners; we have no past faults to condone; no past errors for present criticism. Our errors and mistakes will be manifest during the year; manifest, because we expect to make them; but we do not expect to make the same mistake more than once. We fall back on our human prerogative to excuse an error, but we shall claim no exemption from the consequences of a repetition of the error.

We bespeak for ourselves the help of both active and associate members; with this help we can and will make the JOURNAL a success, without it, we expect to do our best, but the future will have to decide as to our success.

NOTICE.

The Referee desires to announce, that assignments of places on the analysis committees for the present year have been sent out, and he desires to be promptly informed of any failure to receive notice of assignment.

FRITZ H. SMALL,
Referee.

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REPORT OF THE REFEREE FOR 1905.

-:o:-

The Referee on Tannin for 1905 begs to submit the following report.

Early in the year the Referee appointed certain committees with instructions as to the lines of work to be pursued. The committees and subjects are listed below:

1. Evaporation and Drying of Tanning Residues	W. K. Alsop, Chair., H. C. Reed, M. F. Nichols, Chas. Eachus.
2. Soluble Solids Filtration	H. T. Wilson, Chair., M. F. Nichols, A. W. Hoppenstedt, T. J. Mosser.
3. Chroming of Hide Powder	W. K. Alsop, Chair., F. R. Mosbaugh, C. W. Norris, F. H. Small.
4. Analysis of Liquors	F. H. Small, Chair., J. R. Mardick, W. H. Teas, R. P. Cushing.
5. Acid Determination	A. A. Claflin, Chair., F. R. Mosbaugh, C. W. Norris, F. H. Small.
6. Nitrogen Determination	J. H. Yocum, Chair., A. W. Hoppenstedt, J. R. Mardick, A. A. Claflin.
7. Extraction	W. H. Teas, Chair., Chas. Eachus, Geo. A. Kerr, Harry Gerber.
8. Parker-Payne Method of Tannin Analysis	W. H. Teas, Chair., W. N. Holmes, H. T. Wilson, Geo. A. Kerr.
9. Revision of Official Method of Analysis	H. C. Reed, Chair., F. H. Small, W. K. Alsop, W. H. Teas.

Reports have been received from the several committees, with the exception of the Committee on "Acid Determination" and the Committee on "Nitrogen Determination," and are included in, and made a part of, this report.

The report of the committees on "Evaporating and Drying," "Soluble Solids Filtration" and the "Chroming of Hide Powder" having more particularly to do with Study I of the general collaborative work, they are submitted here, as upon the deductions from these reports is based the prospectus of this Study.

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The following suggestions were made to the Chairman of the Committee on "Evaporating and Drying" as to the line of work to be pursued.

1. Tests to prove the relative values of the various methods of evaporation and drying, with especial attention to the combined evaporator and dryer suggested at the last annual meeting of the Association.

2. In connection with the above, the influence of temperature and the length of time of drying upon the Solids, and the uniformity of temperature (and the effect of such upon the Solids) in various portions of the proposed combined evaporator and dryer.

3. The practicability of drying large residues, providing for the contingency that might arise of making up spent tan liquors to required dilution of .35-.45 grams tannin per 100 cc.

The report of the Committee follows:

NEW YORK, May 3rd, 1905.

Mr. H. C. Reed, Referee,
Stamford, Connecticut.

DEAR SIR:

You will find below the analyses submitted by the members of the Committee for the investigation of Evaporating and Drying of Tannin Residues.

Apparently very little work has been done by any member of this Committee, probably owing to lack of time.

HEMLOCK EXTRACT.

Hemlock Ext.	H. C. Reed. Evaporator and Dryer.	W. K. Alsop. Evaporator and Dryer.	M. F. Nichols. Evap. and Dryer.	M. F. Nichols. Water Oven. 97°.	Chas. Eachus. Water Oven. 95°. 5 Hrs.	Chas. Eachus. Water Oven. 95°. 24 Hrs.
Total Solids	53.56	53.34	53.62	53.55	53.55	53.37
Soluble Solids..	51.24	51.08	50.97	51.41	51.40	51.22
Insolubles.....	2.32	2.26	2.65	2.14	2.15	2.15
Non-tannins....	20.58	20.60	20.42	20.53	20.44	20.28
Tannins.....	30.66	30.48	30.55	30.88	30.96	30.94
Oak Ext.						
Total Solids	51.90	51.83	51.93	51.57	52.12	51.44
Soluble Solids..	51.26	51.05	50.72	50.32	51.52	50.76
Insolubles.....	.64	.78	1.21	1.25	.60	.68
Non-tannins....	22.60	22.65	22.21	22.50	22.97	22.62
Tannins.	28.66	28.40	28.51	27.82	28.55	28.14
Chestnut Ext.						
Total Solids	44.37	44.05	44.22	44.73	44.17	44.10
Soluble Solids ..	44.03	43.88	43.85	44.07	43.95	43.89
Insolubles.....	.34	.17	.37	.66	.22	.21
Non-tannins....	12.84	12.84	13.36	13.03	12.80	12.72
Tannins.....	31.19	31.04	30.49	31.04	31.15	31.17

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This work is not as comprehensive as it should be, but it points toward the advisability of a uniform system of evaporation and drying for tannin analyses. The greatest variations in Total Solids occur when the residues were dried in the ordinary form of water oven at temperatures of 95°-97° C., and from 5 to 24 hours. This is true of the Oak and Chestnut extracts, while the results obtained for Hemlock extract agree very well with the solids obtained by the combined evaporator and dryer.

I am strongly in favor of the adoption of a uniform system of evaporation and drying and the combined evaporator and dryer which was described at the last Convention, and is now in use by several members of the Association, seems to be well adapted for the purpose. Attached you will find a sketch and description of one of these dryers. Mr. W. H. Teas says in regard to it, "We are using four combined ovens and dryers, 26" by 28", and they are giving excellent satisfaction. We start the lamps (four Bunsens under each) at 5 p. m., and begin weighing at 8 the next morning, the dishes are as dry at 8 as they are at noon.—— With this system of drying it is possible to secure the highest degree of favorable comparison between the results of chemists in different parts of the country, not only in the ultimate tannin figure, but also in the figures of the different items from which the tannin figure is computed. This, too, at a saving of time, trouble, fuel, change in weight of the dishes, and in the resultant minimum of breakage due to the absence of the necessity for transfer from drier to oven."

Mr. H. C. Reed submits the following tests with this apparatus:

A. TIME TEST.

Elapsed time for evaporation and drying.	Solids grams.
20 hours	.4820
19 "	.4821
18 "	.4820
17 "	.4821
16 "	.4819

B. POSITION TEST.

	Solids grams.
Corner 1	.4816
" 2	.4818
" 3	.4818
" 4	.4819
Side 1	.4819
" 2	.4814
" 3	.4816
" 4	.4816
Middle	.4820

Mr. Reed says further: "The results appear to me to be a very high indorsement of the value of the apparatus, as it would seem impossible to obtain anything but uniformity by its use. Temperature tests show a uniform temperature of 99° C., when taken

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with the bulb about one inch from the plate. Dishes placed on the apparatus in the afternoon are ready for weighing in the morning and much labor is saved by not having to transfer the dishes to a drying oven."

I can indorse all of the statements above, as we have used these ovens for nearly a year with very satisfactory results.

Yours very truly,

W. K. ALSOP.
Chairman of Committee.

(Copy)

Upon the very excellent results from the use of the combined evaporator and dryer, as shown by the foregoing report, is based the method of drying and limits of temperature stated in the prospectus for general work. The Referee's suggested subject 3 was not touched upon.

A recommendation upon evaporation and drying occurs later in this report.

The proposed lines of investigation for the Committee on "Soluble Solids Filtration" were as follows:

1. Influence of temperature upon filtration:

(a) Suggested that solutions be both rapidly and slowly cooled to a definite temperature (say 20° C.), made up immediately, and after pipetting Total Solids, filtration at once proceeded with. Comparisons to be made by this method with present official method. Test to prove whether upon cooling to a definite temperature, the influence of the laboratory temperature has any marked effect upon the resulting Soluble Solids, within the time of filtration.

(b) That solutions be cooled to various temperature and the effect upon the Soluble Solids, of raising the temperature various degrees noted.

NOTE.—In connection with the above, the influence of the cooling upon the Non-tannins must at the same time be determined, thus making an entire analysis imperative.

2. Filtration by means of a filter candle:

The Berkefeld laboratory filter is recommended. Comparative tests with this filter and No. 590, S. & S. 15 cm. paper; also Muncktell's Swedish No. 1 F sheets, cut to 15 cm., size; also

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asbestos filter, layer of asbestos on perforated porcelain plate with suction.

3. Amount of kaolin:

Whether present amount, *viz.*, 2 grams,—is subject to reduction.

Mr. Wilson, Chairman of the Committee submitted the following report:

INSTRUCTIONS GIVEN BY CHAIRMAN TO SOLUBLE SOLIDS COMMITTEE.

1. Influence of temperature upon filtration:

(a) Cool solutions rapidly under hydrant, to 20° C., make up immediately, and after pipetting Total Solids, proceed at once with filtration.

Cool solutions slowly (say in open window) to 20° C., and proceed as above. Comparisons to be made by these methods with the present Official method. Test to prove whether upon cooling to a definite temperature, the influence of the Laboratory Temperature has any marked effect upon the resulting Soluble Solids, within the time of filtration.

(b) Cool solutions to 20° C., and raise at once to 35° C., make up and proceed with analysis. Cool solutions to 10° C., and raise to 20° C. Test to show the effect upon the Soluble Solids of raising the Temperature various degrees after solutions have been cooled.

NOTE.—In connection with (a) and (b), the influence of the cooling of the Non-tannins must at the same time be determined, thus making an entire analysis imperative.

2. Filtration by means of a filter candle. The Berkefeld laboratory filter is recommended. (See Collegium No. 120, page 249),

It is doubtful whether the exact type of filter can be obtained in this country. The Berkefeld filter cylinders can be procured from Eimer and Amend, No. H 2 being most suitable. It may develope that they will not do on account of having metal parts which might be corroded by the tannin solution.

Compare this method with S. & S. No. 590, 15 cm. paper, also Muncktell's Swedish No. 1 F sheets, cut to 15 cm. size; also asbestos filter, layer of asbestos on perforated porcelain plate with suction.

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Use asbestos catalogued as, "washed with nitric acid," and further purify by washing in acid following with water until neutral.

3. Amount of kaolin:

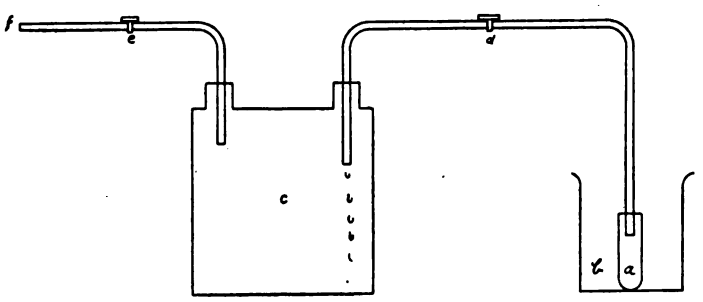
Determine whether present amount, *viz.*, 2 grams, is subject to reduction. Purify your kaolin by washing with dilute HCl following with water until neutral.

Make tests using 2 grams, 1.5 grams, 1 gram.

NOTE.—In all filtrations in 1, 2, and 3, indicate the character of the filtrates, whether or not they are perfectly clear.

It will also be well to make note of the time required for the filtrations by the various methods. Make report on all points as full as possible.

Below is given the method of using the Berkefeld filter.



a—filter candle.

b—beaker.

c—vacuum chamber.

d—stop-cock.

e—stop-cock.

f—to aspirator or vacuum pump.

Fill (*b*) with tannin solution, close (*d*) and exhaust (*c*) as much as possible, then close (*c*) and open (*d*) when the vacuum in (*c*) will draw the liquor from (*b*). To insure a little better vacuum in (*c*) you can leave both (*d*) and (*e*) open until liquor begins to raise in tube, then close (*e*). By this method it is claimed there is absolutely no evaporation.

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To demonstrate whether there is absorption of tannin by the candle, make tests by evaporating the 1st, 2nd, 3rd, and if necessary, the 4th 100 cc. portions of filtrate.

To get at the absorption factor, it will be best to prepare a liter or so of reds free liquor by filtering through paper in large funnel using kaolin. Evaporate 100 cc. of this liquor, then by evaporating successive portions of the filtrate from the candle you can tell exactly how much is required to satisfy the clay before catching the final quantity for the regular analysis.

Subsequent instructions were sent out as follows:

If you have tried your Berkefeld Filter you have probably found the same to be strongly impregnated with iron salts. It will be necessary to remove these before filtering tannin solutions. Boil your filter with 10% hydrochloric acid until iron is removed. This will cause the uncementing of the porcelain mounting, which can be replaced by filling the procelain top with melted paraffine and inserting the cylinder and allowing to cool. Now connect filter with aspirator and draw through water acidified with hydrochloric acid, finishing with distilled water. The filter will now need to be dried before filtering tannin solutions. The reds which adhere to the outside of the filter can be removed with a tooth brush. In filtering, vacuum of three to four inches will be found sufficient. Filter through five portions of 100 cc., and also make Sol. Solids on the same solution using 590 filter paper. To hasten the drying of the filter you can pass through four or five hundred cc. of strong alcohol which will remove the greater part of the water.

TABLE I.

ANALYSES BY PRESENT OFFICIAL METHOD.

CHESTNUT EXTRACT.

	Hoppenstedt.	Mosser.	Nichols.	Wilson.
Temp. of Laboratory ...	19	....	....	25
Time of Filtration.....	....	....	....	75 65
Character of Filtrate ...	Clear	....	....	Brilliant
Total Solids	41.14	44.10[1]	40.81	40.72
Soluble Solids	40.89	43.86[1]	40.38	40.43 40.51
Insoluble Solids.....	.25	.24	.43	.29 .21
Non-tannins	14.44	17.49[1]	14.75	14.26
Tannins	26.45	26.37	25.63	26.17[2] 26.25[3]

[1] Not included in average.
[2] S. & S., 590.
[3] Swedish.

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HEMLOCK EXTRACT.

Temp. of Laboratory...	20	....	....	25
Time of Filtration.....	....	....	....	120 150
Character of Filtrate ...	Clear	....	....	Clear
Total Solids..... ..	48.20	48.72	47.92	48.75
Soluble Solids	45.08	45.35	42.86[1]	45.91 45.17
Insoluble Solids.....	3.12	3.37	5.06[1]	2.84 3.59
Non-tannins	16.13	17.65[1]	14.92	15.25
Tannins	28.95	27.70	27.94	30.66[2] 29.92[3]

[1] Not included in average.
[2] S. & S., 590.
[3] Swedish.

TABLE I[a].

ANALYSES BY PRESENT OFFICIAL METHOD.

SOLID QUEBRACHO EXTRACT.

Temp. of Laboratory ...	20.5	....	....	25
Time of Filtration.....	....	....	....	120 90
Character of Filtrate ...	Clear	....	....	Clear
Total Solids..	78.69	78.58	78.98	78.62
Soluble Solids.....	71.38	74.00[1]	71.45	71.69 71.75
Insoluble Solids.....	7.31	4.58[1]	7.53	6.93 6.87
Non-tannins	10.30	13.36[1]	10.83	10.65
Tannins	60.58	60.64	60.62	61.04[2] 61.10[3]

[1] Not included in average.
[2] S. & S., 590.
[3] Swedish.

AVERAGES.

	Total Solids.	Soluble Solids.	Insoluble Solids.	Non-Tannins.	Tannins.
Chestnut Extract.					
Average.....	40.89	40.55	.28	14.48	26.17
Maximum	41.11	40.89	.43	14.75	26.45
Minimum.....	40.72	40.38	.21	14.26	25.63
Greatest Dif. from Avg	0.25	0.35	.15	0.27	0.54
Hemlock Extract.					
Average	48.39	45.38	3.23	15.43	29.03
Maximum	48.75	45.91	3.59	16.13	30.66
Minimum	47.92	45.08	2.84	14.92	27.70
Greatest Dif. from Avg	0.47	0.53	0.39	0.70	1.63
Solid Quebracho Extract.					
Average	78.72	71.57	7.16	10.76	60.79
Maximum	78.98	71.75	7.53	10.83	61.10
Minimum.....	78.58	71.38	6.87	10.65	60.58
Greatest Dif. from Avg	0.26	0.19	0.29	0.11	0.31

TABLE II.

Solutions Cooled Quickly to 20° in Water at 15°.

CHESTNUT EXTRACT.

	Hoppenstedt.	Mosser.	Nichols.	Wilson.
Temp. of Solution.....	20	....	20	20
Time of Filtration.....	....	....	....	90 80
Character of Filtrate ...	Clear	....	....	Brilliant
Total Solids	41.07	....	40.56	40.73
Soluble Solids	40.94	....	40.25	40.44 40.63
Insoluble Solids.....	.13	....	.31	.29 .10
Non-tannins	14.45	....	13.97	14.52
Tannins	26.49	....	26.28	25.92[1] 26.11[2]

[1] S. & S., 590.
[2] Swedish.

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Solutions Cooled Quickly to 20° in Water at 15°.

HEMLOCK EXTRACT.

Temp. of Solution.....	20	20	20	20	
Time of Filtration.....	....	....	....	230	215
Character of Filtrate ...	Clear	....	....	Brilliant	
Total Solids.....	48.36	47.78	48.35	48.82	
Soluble Solids	45.04	45.67	45.21	45 02	45.02
Insoluble Solids.... ...	3.32	2.11	3.14	3.80	3.80
Non-tannins	16.60	16.94	15.39	15.73[2]	
Tannins	28.44	28.73[1]	29.82	29.29[3]	29.29[4]

[1] Cooled under hydrant.
[2] Slight cloud.
[3] S. & S., 590.
[4] Swedish.

TABLE II[a].

Solutions Cooled Quickly to 20° in Water at 15°.

SOLID QUEBRACHO EXTRACT.

	Hoppenstedt.	Mosser.	Nichols.	Wilson.	
Temp. of Solution.....	20	20	20	20	
Time of Filtration.....	....	....	....	90	85
Character of Filtrate ...	Clear	....	....	Clear	
Total Solids.....	79.08	79.01	78.63	79.65	
Soluble Solids.....	71.85	72.88	71.06	73.95	73.87
Insoluble Solids.....	7.23	7.13	7.57	5.70	5.78
Non-tannins	10.98	12.63[1]	10.10	10.73	
Tannins	60.87	60.25[1]	60.96	63.22[2]	63.14[3]

[1] Cooled under hydrant.
[2] S. & S., 590.
[3] Swedish.

AVERAGES.

	Total Solids.	Soluble Solids.	Insoluble Solids.	Non-Tannins.	Tannins.
Chestnut Extract.					
Average	40.79	40.56	0.21	14.31	26.20
Maximum	41.07	40.94	0.31	14.52	26.49
Minimum.....	40.53	40.25	0.10	13.97	25.92
Greatest Dif. from Avg	0.28	0.38	0.11	0.34	0.29
Hemlock Extract.					
Average	48.33	45.19	3.23	16.16	29.11
Maximum	48.82	45.67	3.80	16.94	29.82
Minimum.....	47.78	45.02	2.11	15.39	28.44
Greatest Dif. from Avg	0.55	0.48	0.69	0.78	0.71
Solid Quebracho Extract.					
Average	79.09	72.72	6.48	11.11	61.69
Maximum	79.65	73.95	7.57	12.63	63.22
Minimum.....	78 63	71.06	5.70	10.10	60.25
Greatest Dif. from Avg	0.56	1.66	1.09	1.52	1.53

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TABLE III.

Solutions Cooled Slowly in Open Window.

CHESTNUT EXTRACT.

	Hoppenstedt.	Mosser.	Nichols.	Wilson.
Temp. of Solution	20	....	20	20[1]
Time of Filtration	....	....	...	60 60
Character of Filtrate	Clear	....	....	Brilliant
Total Solids	41.18	....	40.25	41.02
Soluble Solids	41.02	....	40.00	40.46 40.46
Insoluble Solids	.16	....	.25	.56 .56
Non-tannins	14.64	....	14.40	14.46
Tannins	26.38[2]	....	25.60	26.00[3] 26.00[4]

[1] Time of cooling, 2 hours.
[2] Outside temp., 5°.
[3] S. & S., 590.
[4] Swedish.

Solutions Cooled Slowly in Open Window.

HEMLOCK EXTRACT.

Temp. of Solution	20	20	20	20[2]
Time of Filtration	....	....	....	180 215
Character of Filtrate	Clear	....	....	Brilliant
Total Solids	48.40	48.35	47.85	48.80
Soluble Solids	45.33	46.18	45.64	45.08 45.00
Insoluble Solids	3.07	2.17	2.21	3.72 3.80
Non-tannins	16.59	....	15.39	15.20
Tannins	28.74[1]	....	30.25	29.88[3] 29.80[4]

[1] Outside Temp., 5°.
[2] Time of cooling, 2 hours.
[3] S. & S., 590.
[4] Swedish.

TABLE III[a].

Solutions Cooled Slowly in Open Window.

SOLID QUEBRACHO EXTRACT.

	Hoppenstedt.	Mosser.	Nichols.	Wilson.
Temp. of Solution	20	20	20	20
Time of Filtration	....	....	....	70 70
Character of Filtrate	Clear	....	....	Brilliant
Total Solids	79.08	78.48	79.24	79.18
Soluble Solids	71.69	76.59	71.67	72.81 72.69
Insoluble Solids	7.39	1.89	7.57	6.37 6.49
Non-tannins	11.17	....	10.45	10.67
Tannins	60.52[1]	....[2]	61.22	62.14[3] 62.02[4]

[1] Outside temp., 5°.
[2] Not included in average.
[3] S. & S., 590.
[4] Swedish.

AVERAGES.

	Total Solids.	Soluble Solids.	Insoluble Solids.	Non-Tannins.	Tannins.
Chestnut Extract.					
Average	40.81	40.48	0.38	14.50	25.99
Maximum	41.18	41.02	0.56	14.64	26.38
Minimum	40.25	40.00	0.16	14.40	25.60
Greatest Dif. from Avg	0.56	0.48	0.22	0.14	0.39

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AVERAGES.—Continued.

	Total Solids.	Soluble Solids.	Insoluble Solids.	Non-Tannins.	Tannins.
Hemlock Extract.					
Average	48.35	45.40	2.97	15.72	29.67
Maximum	48.80	46.18	3.80	16.59	30.25
Minimum	47.85	45.00	2.17	15.20	28.74
Greatest Dif. from Avg	0.50	0.78	0.83	0.87	0.93
Solid Quebracho Extract.					
Average	79.16	72.21	6.95	10.76	61.47
Maximum	79.24	72.81	7.57	11.17	62.14
Minimum	79.08	71.67	6.37	10.45	60.52
Greatest Dif. from Avg	0.08	0.60	0.62	0.41	0.95

TABLE IV.

Solutions Cooled Quickly to 20° in Water at 15°, Then Warmed to 35°.

CHESTNUT EXTRACT.

	Hoppenstedt.	Mosser.	Nichols.	Wilson.
Temp. of Solution	35	....	35	35
Time of Filtration	....	....	....	60
Character of Filtrate	Clear	....	....	Brilliant
Total Solids	41.13	....	41.06	40.88
Soluble Solids	41.13	....	40.81	40.75
Insoluble Solids	.00	....	.25	.13
Non-tannins	14.59	....	14.18	14.84
Tannins	26.54[1]	....	26.63	25.91

[1] Laboratory temperature, 19°.

Solutions Cooled Quickly to 20° in Water at 15°, Then Warmed to 35°.

HEMLOCK EXTRACT.

Temp. of Solution	35	35	35	35
Time of Filtration	....	....	....	80
Character of Filtrate	Slight cloud	....	....	Clear
Total Solids	48.42	47.95	48.28	48.70
Soluble Solids	47.21	46.10	46.57	47.64
Insoluble Solids	1.21	1.85	1.71	1.06
Non-tannins	16.19	17.14	15.45	15.45
Tannins	31.02[1]	28.96[2]	31.12	32.19

[1] Laboratory temperature, 20°C.
[2] Cooled under hydrant.

TABLE IV[a].

Solutions Cooled Quickly to 20° in Water at 15°, Then Warmed to 35°.

SOLID EXTRACT QUEBRACHO.

	Hoppenstedt.	Mosser.	Nichols.	Wilson.
Temp. of Solution	35	35	35	35
Time of Filtration	....	....	....	60
Character of Filtrate	Clear	....	....	Brilliant
Total Solids	78.92	77.75[2]	79.39	79.29
Soluble Solids	72.46	71.66	72.42	73.81
Insoluble Solids	6.46	6.09	6.97	5.48
Non-tannins	10.98	12.00[2]	10.80	10.84
Tannins	61.48[1]	[2]59.66[3]	61.62	62.97

[1] Labratory Temperature, 20.5°.
[2] Not included in average.
[3] Cooled under hydrant.

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AVERAGES.

	Total Solids.	Soluble Solids.	Insoluble Solids.	Non-Tannins.	Tannins.
Chestnut Extract.					
Average	41.02	40.89	0.12	14.54	26.36
Maximum	41.13	41.13	0.25	14.84	26.63
Minimum	40.88	40.75	0.00	14.18	25.96
Greatest Dif. from Avg	0.14	0.24	0.13	0.36	0.40
Hemlock Extract.					
Average	48.34	46.88	1.45	16.05	30.83
Maximum	48.70	47.64	1.85	17.14	32.19
Minimum	47.95	46.10	1.06	15.45	28.65
Greatest Dif. from Avg	0.39	0.78	0.40	1.09	1.87
Solid Quebracho Extract.					
Average	79.20	72.59	6.25	10.87	62.02
Maximum	79.39	73.81	6.97	10.98	62.97
Minimum	78.92	71.66	5.48	10.80	61.48
Greatest Dif. from Avg	0.28	1.22	0.77	0.11	0.95

TABLE V.

Solutions Cooled Quickly to 10° in Water at 8°, Then Warmed to 20°.

CHESTNUT EXTRACT.

	Hoppenstedt.	Mosser.	Nichols.	Wilson.
Temp. of Solution	20	....	20	20
Time of Filtration	....	....	....	160
Character of Filtrate	Clear	....	....	Brilliant
Total Solids	41.13	....	40.56	40.90
Soluble Solids	40.98	....	40.25	40.45
Insoluble Solids	.15	....	.31	.45
Non-tannins	14.49	....	14.33	14.57
Tannins	26.49[1]	....	25.92	25.88

[1] Laboratory temperature, 19°.

Solutions Cooled Quickly to 10° in Water at 8°, Then Warmed to 20°.

HEMLOCK EXTRACT.

Temp. of Solution	20	20	20	20
Time of Filtration	....	....	....	300
Character of Filtrate	Clear	....	....	Brilliant
Total Solids	48.33	48.00	47.00[2]	48.78
Soluble Solids	45.09	45.53	43.57[2]	44.84
Insoluble Solids	3.24	2.47[2]	3.43	3.94
Nan-tannins	16.10	18.51[2]	14.17[2]	15.45
Tannins	28.99[1]	27.02[2]	29.40	29.39

[1] Laboratory temperature, 20°.
[2] Not included in average.

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TABLE V[a].

Solutions Cooled Quickly to 10° in Water at 8°, Then Warmed to 20°.

SOLID QUEBRACHO EXTRACT.

	Hoppenstedt.	Mosser.	Nichols.	Wilson.
Temp. of Solution	20	....	20	20
Time of Filtration	....	....	....	150
Character of Filtrate	Clear	....	....	Brilliant
Total Solids	79.08	....	79.24	79.97
Soluble Solids	71.23	....	71.36	74.52[2]
Insoluble Solids	7.85	....	7.88	5.45[2]
Non-tannins	10.89	....	10.45	11.00
Tannins	60.34[1]	....	60.91	63.52[2]

[1] Laboratory temperature, 20.5°.
[2] Not included in average.

	Total Solids.	Soluble Solids.	Insoluble Solids.	Non-Tannins.	Tannins.
Chestnut Extract.					
Average	40.86	40.56	0.30	14.46	26.09
Maximum	41.13	40.98	0.45	14.57	26.49
Minimum	40.56	40.25	0.15	14.33	25.88
Greatest Dif. from Avg	0.30	0.42	0.15	0.13	0.40
Hemlock Extract.					
Average	48.37	45.15	3.54	15.77	29.26
Maximum	48.78	45.53	3.94	16.10	29.40
Minimum	48.00	44.84	3.24	15.45	28.99
Greatest Dif. from Avg	0.41	0.38	0.40	0.33	0.27
Solid Quebracho Extract.					
Average	79.43	71.29	7.86	10.78	60.62
Maximum	79.97	71.36	7.88	11.00	60.91
Minimum	79.08	71.23	7.85	10.45	60.34
Greatest Drif. fom Avg	0.54	0.13	0.02	0.33	0.29

TABLE VI.

INFLUENCE OF AMOUNT OF KAOLIN UPON FILTRATION.

		Chestnut.			Hemlock.			Quebracho.		
		T. S.	S. S.	R.	T. S.	S. S.	R.	T. S.	S. S.	R.
Hoppenstedt.										
2gms.	590 1..	41.11	40.99	.12	48.40	44.75	3.65	79.54	72.23	7.31
2 "	Sw. 2..	41.11	40.80	.31	48.40	44.71	3.69	79.54	71.92	7.62
1.5 "	3..	41.11	40.99	.12	48.40	45.14	3.26	79.54	72.31	7.23
1.0 "	4..	41.11	40.96	.15	48.40	45.35	3.05	79.54	72.31	7.23
		All filtered in 1 hour.			All filtered in 5½ hours. All filtrates clear.			1 and 2—4 hrs. 3 and 4—5½ hrs.		
Wilson.										
2 gms.	590.....	40.58	39.83	.75	48.72	44.65	4.07	79.45	71.67	7.78
2 "	590.....	40.58	39.85	.73	48.72	44.75	3.97	79.45	....	...
1.5 "	590.....	40.58	39.94	.64	48.72	44.78	3.94	79.45	71.75	7.70
1.0 "	590.....	40.58	39.95	.63	48.72	44.69	4.03	79.45	71.86	7.59
		1½ hours.			3½ hours. All filtrates clear.					
Mosser.										
2 gms.	590							79.01	73.63	5.38
2 "	Sw							79.01	73.33	5.68
1.5 "	590							79.01	72.72	6.29
1.0 "	590							79.01	74.00	5.01
Sw.- Swedish								4 hours.		

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TABLE VI[a].

INFLUENCE OF AMOUNT OF KAOLIN UPON FILTRATION.

HEMLOCK EXTRACT.

Nichols.	Total Solids.	Soluble Solids.	Reds.
2 gms.	48 00	42.00	6.00
		41.28	6.72
1.5 "	48.00	41.43	6.57
		41.07	6.93
1 "	48.00	42.00*	6.09
		41.43	6 57

* All clear except *.

COMPARISON OF FILTER PAPER.

	Chestnut.		Hemlock.		Quebracho.	
Nichols.	Swedish.	590	Swedish.	590	Swedish.	590
Total Solids.....	41.06	41.06.	48.50	48.50	79.24	79.24
Soluble Solids.....	40.75	40 94	43.93	43.50	69.70	70.61
Insoluble Solids.....	.31	.12	4.57	5.00	9.54	8.63
Time	1½ hrs.	1½ hrs.	6 hrs.	6 hrs.	6 hrs.	6 hrs.

TABLE VII.

Precipitation of Reds in Chestnut Extract.

Solutions Made at 80°, Cooled Quickly to 20° in Water at 15° and Diluted to Official Strength, with Water at 20°.

Test to show that reds once precipitated in a strong Solution are not immediately redissolved upon dilution with Cold Water.

FILTERED DIRECT.

Wilson.	Official				
Barkometer	3.2°	8°	15°	20°	30°
Total Solids.....	41.07	40.85	40.95	40.87	40.97
Soluble Solids.....	40.94	40.45	40.37	40.35	40.32
Insoluble Solids.....	0.13	0.40	0.58	0.52	0.65
DILUTIONS.					
Total Solids.....	....	40.84	40.80	40.70	41.02
Soluble Solids.....	....	40.44	40.38	40.21	40.33
Insoluble Solids.....	....	0.40	0.42	0.49	0.69
FIGURES ON DILUTION BY MR. HOPPENSTEDT.					
Barkometer	3.5°	10°	19°	28.5°	....
Total Solids.....	44.59	44.51	44.55	44.58	....
Soluble Solids.....	44.24	44.20	43.85	43.88	....
Insoluble Solids.....	0.35	0.31	0.70	0.70	....

TABLE VIII.

THE BERKEFELD FILTER CANDLE.

Wilson.	Time Vac.	Chestnut.	Time vac.	Hemlock.	Time vac.	Quebracho.
1st 100 cc.....	13m/2″3″ B.	40.30	..... C.	....	7m/3-4 B.	72.70
2nd "	10m/3″4″ "	40.38	..... SC.	....	6m/4-5 "	72.71
3rd "	7m/5″ "	40.36	26m/4″5″ B	44.92	11m/4-5 CL.	72.72
4th "	7m/6″ "	40.32	11m/2″3″ CL.	45.10	22m/5-6 SC.	72.89
5th "	8m/6″ "	40.25	20m/5″6″ B.	44.94	....	....
6th "	10m/7″ "	40.24	30m/6″10″ CL.	45.04	....	.....

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THE BERKEFELD FILTER CANDLE.—Continued.

	Time Vac.	Chestnut.	Time Vac.	Hemlock.	Time Vac.	Quebracho.
Average	....	40.31	.....	45.00	.....	72.75
S. & S.—590	60m	" 40.37	146m	SC. 45.29	100m B.	71.00
T. S	....	40.86	.....	48.63	.....	78.79
Reds by candle.	....	.55	.....	3.63	.....	6.04
Reds by 590	....	.49	.....	3.34	.....	7.79
Mosser.						
T. S	....	....	.....	48.24	.....	79.01
S. S	....	....	60m	CL. 45.13	60m CL.	72.78
Insol	....	....	.....	3.11	.....	6.23

B.—Brilliant.
C.—Cloudy.
SC.—Slight Cloud.
CL.—Clear.

TABLE VIII[a].

Filtrations with Berkefeld Filter Candle on Reds Free Chestnut Liquor.

Test to show whether or not there is any absorption or evaporation.

Wilson.		
Total Solids	40.35	40.47
1st 100 cc	39.87	39.86
2nd "	40.38	40.36
3rd "	40.35	40.45
4th "	40.32	40.47
5th "	40.32	40.46
Average 2-5th	40.34	40.43

No absorption after 1st 100 cc. and apparently no evaporation.

TABLE IX.

FILTRATIONS BY ASBESTOS FILTER.

Wilson.	Chestnut.	Hemlock.[1]	Quebracho.[1]
Total Solids	40.86	48.50	78.79
1st 100 cc	39.80	46.20	73.22
2nd "	40.34	47.98	73.08
3rd "	40.62	47.96	73.49
4th "	40.71	47.90	73.94

[1] All very cloudy.

COMPARISON OF AVERAGES—TABLES I-V.

CHESTNUT EXTRACT.

	Table I.	Table II.	Table III.	Table V.	Table IV.
Total Solids	40.89	40.79	40.81	40.86	41.02
Soluble Solids	40.55	40.56	40.48	40.56	40.89
Insoluble Solids	0.28	0.21	0.38	0.30	0.12
Non-tannins	14.48	14.31	14.50	14 46	14.54
Tannins	26.17	26.20	25.99	26.09	26.36

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CHESTNUT EXTRACT—Continued.

	Table I.	Table II.	Table III.	Table V.	Table IV.
Hemlock Extract.					
Total Solids	48.39	48.33	48.35	48.37	48.34
Soluble Solids	45.38	45.19	45.54	45.15	46.88
Insoluble Solids	3.23	3.23	2.97	3.54	1.45
Non-tannins	15.43	16.16	15.72	15.77	16.05
Tannins	29.03	29.11	29.67	29.26	30.83
Solid Quebracho Extract.					
Total Solids	78.72	79.09	79.16	79.43	79.20
Soluble Solids	71.57	72.72	72.21	71.29	72.59
Insoluble Solids	7.16	6.48	6.95	7.86	6.25
Non-tannins	10.76	11.11	10.76	10.78	10.87
Tannins	60.79	61.69	61.47	60.62	62.02

SOLUBLE SOLIDS.

INFLUENCE OF TEMPERATURE UPON FILTRATION.

A. Extract dissolved in the Official way.

B. Extract solution by rapidly cooling to 20 C., with water at 15 C.

OAK EXTRACT NO. 1.

Hoppenstedt.

	A.	B.
Moisture	48.53	48.40
Total Solids	51.47	51.60
Soluble Solids	50.98	51.32
Reds	.49	.28
Non-tannins	23.30	23.26
Tannins	27.68	28.06

OAK EXTRACT NO. 2.

	A.	B.
Moisture	52.21	52.34
Total Solids	47.79	47.66
Soluble Solids	47.29	47.16
Reds	.50	.50
Non-tannins	.50	.50
Tannins	25.91	25.87

OAK EXTRACT NO. 3.

	A.	B.
Moisture	51.50	51.30
Total Solids	48.50	48.62
Soluble Solids	48.08	48.13
Reds	.42	.49
Non-tannins	21.77	21.85
Tannins	26.31	26.28

OAK EXTRACT NO. 4.

	A.	B.
Moisture	49.82	49.93
Total Solids	50.18	50.07
Soluble Solids	49.19	49.51
Reds	.99	.56
Non-tannins	22.14	22.13
Tannins	27.05	27.38

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SOLID QUEBRACHO EXTRACT. (ORDINARY).

	A.	B.
Moisture	24.75	24.50
Total Solids	75.25	75.50
Soluble Solids	69.17	69.17
Reds	6.08	6.33
Non-tannins	6.98	7.08
Tannins	62.19	62.09

SOLID QUEBRACHO EXTRACT. (CLARIFIED).

	A.	B.
Moisture	24.17	24.00
Total Solids	75.83	76.00
Soluble Solids	75.00	75.67
Reds	.83	.33
Non-tannins	13.77	14.06
Tannins	61.23	61.61

THE BERKEFELD FILTER.

Filtrations on Tannery Liquors.

S. & S. 590 paper and filter candle compared.

The entire series filtered through one candle by simply rejecting the first 125 cc. of each liquor.

Liquors alternately fresh and sap.

Time — 590 = 1 hr. 40 min. Candle = 1 hr. 45 min.

Counting intermissions, or 10½ min., for each filtration of 250 cc.

Vacuum = 9 — 11 inches.

Wilson.

	Barkometer.	S. &. S. 590.	Candle.	Diff.
Rocker	19.30	4.26	4.31	+ .05
New	39.80	9.28	9.36	+ .08
Sap	31.30	7.42	7.47	+ .05
New	33.80	7.89	7.82	— .07
Sap	29.30	7.25	7.27	+ .02
New	32.80	7.81	7.80	— .01
Sap	29.30	7.02	7.05	+ .03
New	33.30	7.77	7.84	+ .07
Sap	32.80	7.71	7.78	+ .06
Leaches	43.80	10.74	10.88	+ .14

CHAIRMAN'S REPORT UPON SOLUBLE SOLIDS.

Mr. H. C. Reed, Referee.

DEAR SIR:

In outlining the work on Soluble Solids for the present year, the chairman has followed the suggested line laid out by the Referee, incorporating such points as were suggestd by members of the Committee.

The outline was sent out early in the year and although lengthy, we do not think it covered too much ground to be handled by each

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member of the Committee. We therefore regret that some members found the task so great as to fall short of a complete report.

In the hope of securing uniformity of results, large samples of three extracts were sent out to each member with instructions sufficiently explicit to meet all requirements.

In spite of the fact that all were working, presumably on the same basis, exactly, the figures when compared are far from settling the question of uniformity and bring us to the point where we arrive each year. The question then again arises what shall we do to secure uniformity? There are many things which labor against this end, of which, personal error, temperature, time, method of drying residues and influence of filter paper, play most important part. It is extremely difficult to bind a large number of people to every point of a set rule. Here comes the personal error. One man will depart one way ever so little perhaps and the next man to the same degree in another direction in some point of lesser importance. In the end, however, these slight deviations may become the prime factor in the non-concordance of results. Such things have come under our own observation, and this is why it is mentioned. At the present day commercial chemistry finds its way into almost every great line of industry and the same thing which is true in our case is true in other branches of the profession, namely, lack of agreement in comparative work. There are vast amounts of money at stake on the findings of the commercial chemists, and the constant disagreements of those in the same line of work leave a bad impression of the profession upon the unlearned. Therefore, anything that we can do in our line towards getting to gether will be so much gained. In our report this year there is some good individual work which goes to show that there could be equally good collective work, all things being equal at the start.

2nd. Temperature is a point which must be defined in our method. A review of the work done this year on this line proves it to be of prime importance, but at the same time very difficult to define. How are we to compel a man in Florida, for instance, to work at the same temperature as a man in Canada? In a country so large and of such diversified temperature this is a hard question to answer. Yet in spite of this very apparent obstacle, it is up to us to confine the analyses of commercial products to as narrow a

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range of temperature as possible. Some favor the plan of cooling the solutions quickly to a definite temperature, in water at a definite temperature and proceeding at once with analyses. When one has a large number of analyses to get out it is difficult to pursue this course, and get all of the work out of the way during working hours. It seems to us that a slight modification of this plan as suggested by the Referee would meet our requirements better, *i. e.*, make up as per Official Method in the evening and in the morning, bring to a definite temperature by either warming or cooling with water at a stated temperature. By this method change of temperature would be small and it would at the same time give one a good start on the day's work.

Mr. Hoppenstedt has done considerable satisfactory work on cooling solutions to 20° in water at 15°, as compared with the Official Method. On the average the Official Method gave him higher Insolubles. This also holds true in the comparative work of the Committee. There is a constant decrease in the Insolubles as the temperature is raised to 35°, hemlock being the most susceptible to temperature changes. As shown in the tables, the Insolubles are still subject to change of temperature even after analysis solution has been cooled to 20° in water at 15°. Here we come to an interesting set of figures, Table VII, Precipitation of Reds, a line suggested by Mr. Hoppenstedt, but unfortunately not fully carried out by him. The chairman made up five solutions of chestnut extract, beginning with Official strength and going up to 8°, 15°, 20°, and 30° Barkometer. The extract was dissolved with water at 80° and cooled to 20°, in water at 15°. Dilutions to Official strength of the four stronger solutions were then made by diluting an aliquot part with water at 20°. Both the strong and the dilute solutions were filtered through paper, with results showing that the Insolubles once precipitated in a strong solution are not immediately redissolved upon dilution to Official strength with cold water. Something on this line was mentioned by Mr. Claflin in his remarks at the St. Louis convention, and if it should be borne out by further investigation, then we have a straw to cling to in the Soluble Solids sea.

3rd. Time is another factor which has its direct bearing upon the question in hand. Its influence is two-fold. The length of time the solution stands before analysis and the time of filtration.

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It has been observed by the writer that a solution standing for twenty-four hours will show more Reds than one standing half that time. In filtration, time is important both in regard to evaporation and filter paper absorption. An accurate means of rapid filtration would go far towards solving these problems, as well as those of temperature.

4th. Although the methods of drying tannin residues as practiced at the present time may not exert any great influence upon the results, yet the influence exists. Tannin residues are not stable at high temperatures. There has been a great deal of work done in the past on this subject and a method drawn up to cover drying by various means. Nevertheless, in view of the fact that drying has its influence, we believe that on the analysis of commercial products, everyone should use the same kind of drying apparatus. Some dry at 98° in a water oven, others at 103° to 105° in air-bath. Would it be gonig too far to require either the one or the other? When one is doing factory work, personal error, temperature, time, method of drying, etc., do not matter so much so long as one always does the work in the same way, but when it comes to strictly compartive work, participated in by many, the methods of working should be similar.

5th. One of the most annoying features in the analysis of tannins is the filtrations for Soluble Solids. Various well-known means have been devised to obviate the error due to filter paper absorption. So far all have fallen short of the objective point and are likely to continue to do so, so long as we are dealing with a substance which has no definite stopping place in its capacity for absorbing tannins. If every filter had the same absorption value the problem would be an easy one, but even the best papers obtainable vary in thickness and texture to a remarkable degree. Here I would call your attention to Table VII, previously alluded to on page 3, which may show at least a partial solution of this difficulty. Of the 30° Barkometer solution, filtered direct, only the first 25 or 30 cc. were rejected. Yet the Soluble Solids figure as identical to that obtained on the diluted solution, of which 150 cc. were rejected. By employing a stronger solution for the Soluble Solids and Insolubles figures the filter paper would be more quickly satisfied, giving us at the same time truer figures in both items. Of the two papers experimented with this year there

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seems to be little choice except that the No. 590 comes already cut in circles.

During the experimental work on Soluble Solids last year the writer tried various methods of filtrations in the attempt to get around the filter paper difficulty. Asbestos filters of various forms, sand filters of various forms, combination of asbestos and sand and parchmentized paper pulp, were all given a thorough test, but with purely negative results. This year the asbestos filter was incorporated in the outline for the Committee, but none of the collaborators returned any figures on same. The writer obtained the purest asbestos fiber on the market, which was subjected to further purification in the laboratory. From this, filters of closely packed fiber, at least ½" in thickness, were prepared with the utmost care, with results as given in Table IX. The first 40 or 50 cc. passing through the filter are brilliantly clear, but robbed of tannin. After a short time the Reds work their way through and the results rapidly approach the Total Solids figure. It appears then that we must continue to put up with what we know to be an imperfect method and continue our search for something better.

6th. The figures on Insolubles obtained by using varying quantities of kaolin show a very slight decrease as the amount of the latter is lessened. The differences are so slight that the chairman would not recommend a change in the present quantity, especially since filtrations with one gram are slightly slower than when the regular quantity is used. Two grams of well purified kaolin does not seem to have any harmful influence.

7th. The entirely new departure in the work this year is the filtration of tannin solutions by the Berkefeld filter candle. The candles as they appear on the market are not immediately fit for use on Tannin solutions, but must be previously boiled in dilute hydrochloric acid to remove iron and other soluble salts. Messrs. Parker and Payne, of the I. A. L. T. C., have obtained some very remarkable results by the use of this filter and have strongly recommended to their association, the adoption of same. With the above in mind the chairman entered upon the work this year in the hope that at least there was in sight a panacea for our Soluble Solids ills. The results, however, were only partially encouraging. It is to be very much regretted that the collaborators through stress of work or other reasons, were not able to turn in a com-

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plete set of figures upon this point. The chairman was very successful both in obtaining good results and in the manipulation of the filter. However, his work is almost entirely lacking in corroboration by other members of the association, and hence are of small weight. It is nevertheless our opinion that the work is sufficiently worthy to merit further and fuller investigation, and we recommend to you that such be done. The filter is rapid, gives brilliant filtrates, eliminates evaporation, is non-absorptive after the first 50 or 100 cc., is inexpensive and not difficult to manipulate.

Having sincerely endeavored to make our tables and report to you as full and complete as possible, we trust that the same will at least partially meet with your approval.

Very truly yours,

H. T. WILSON,
Chairman Soluble Solids Com.

The result of the Committee's work was neither as satisfactory nor conclusive as desired. The Referee therefore deviated in his outline of general collaborative work from the methods pursued by the committee, and followed one which occurred to him later and is mentioned in the chairman's report, *viz.*, methods for Soluble Solids as per Study I (b).

Recommendations pertaining to this subject are found later in this report.

The suggestions offered to the Chairman of the Committee on the "Chroming of Hide Power" were as follows:

1. Determination of the best compound of Chromium for chroming purposes, to determine whether the sulphate factor cannot be eliminated.

2. Influence of time in chroming, determining whether the time as per present official method cannot be shortened, and whether all the chroming compound cannot be added at once instead of in two portions as now obtains.

3. Determination as to whether the amount of hide powder used for detannizing as per present official method, cannot be reduced.

4. Influence of the acidity or alkalinity of hide powder upon the chroming and upon the resulting Non-tannins.

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5. Effect of greater or less moisture in the wet hide powder upon the resulting Non-tannins.

6. Whether the test for tannin in the non-tannin filtrate as per present official method cannot be advantageously altered.

7. The question of the production of a dry chromed hide powder, and tests with a hide powder made by grinding dry chromed hide powder.

Mr. Alsop, Chairman of the Committee, submitted the following report:

NEW YORK, June 14th, 1905.

Mr. H. C. Reed, Referee,
Stamford, Connecticut.

DEAR SIR:

I beg to submit the report for the Committee appointed to investigate the "Chroming of Hide Powder." This Committee consisted of Messrs. C. C. Hildebrand, F. R. Mosbaugh, H. C. Reed, and W. K. Alsop, Chairman. Four samples of extracts, Liquid Quebracho, Chestnut, Hemlock and Oak, were sent out together with a request for certain experiments. Below you will find tabulations of such results as are deemed to be of sufficient importance to report.

No. 1. Chroming Hide Powder.

1. Chroming Hide Powder as called for by the Official method.

2. Chroming Hide Powder as called for by the Official method, except in one particular; add all the Chrome at once instead of in two portions.

3. Chrome the Powder as in No. 2, except that as soon as the Chrome is added agitate by either stirring or by shaking for (1) one-half hour, (2) for one hour.

1. Official.	Wt. Wet Hide. Grams.	Per ct Water in Wet Hide	Dry Hide. Gms.	Per Cent. Non-tannins. Chestnut.	Hemlock.	Oak.	Quebracho.	
Reed.....	46	72.83	12.50	12.84	20.58	22.60	7.63	
Hildebrand..	50	78.50	10.75	13.51	20.90	23.17	8.69	
Mosbaugh...	40	63.00	14.80	12.51	20.18	22.35	7.75	Hide No. 1
Mosbaugh...	40	65.00	14.00	12.78	20.25	22.27	7.91	Hide No. 2
Alsop.....	50	72.25	13.87	12.84	20.60	22.65	7.87	
2. Chrome added all at once.								
Reed.....	44	71.02	12.75	12.96	20.52	22.68	7.72	
Hildebrand .	46	71.60	12.88	13.25	20.60	23.04	7.91	
Mosbaugh...	40	65.00	14.00	12.80	20.25	22.66	7.91	Hide No. 1
Mosbaugh...	40	65.00	14.00	12.88	20.42	22.58	8.14	Hide No. 2
Alsop.....	50	74.00	13.00	12.85	20.41	22.68	7.67	

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1. Official.	Wt. Wet Hide. Grams.	Per ct. Water in Wet Hide	Dry Hide Gms.	Per Cent. Non-tannins. Chest-nut.	Hem-lock.	Oak	Que-bracho.	
3. Chromed ½ hour.								
Reed.....	42.5	72.00	11.90	13.42	20.94	23.18	8.16	
Hildebrand..	50	75.50	12.25	13.50	20.79	22.94	8.26	
Mosbaugh...	40	64.00	14.40	13.10	20.63	22.90	8.46	Hide No. 1
Mosbaugh...	40	63.00	14.80	13.32	20.64	22.55	8.31	Hide No. 2
Alsop.....	51	74.00	13.26	13.37	20.92	22.84	8.19	
3. (2) Chromed 1 hour.								
Reed.....	46	73.50	12.20	13.08	20.72	22.71	7.70	
Hildebrand..	46	72.00	12.88	13.51	20.02	23.39	8.65	
Mosbaugh...	40	64.00	14.40	12.95	20.87	22.80	8.20	Hide No. 1
Mosbaugh...	40	62.75	14.90	13.00	20.56	22.59	8.09	Hide No. 2
Alsop.....	51	72.50	14.03	13.15	20.72	22.53	8.24	
4. Unchromed.								
Reed.....	48	73.40	12.77	13.61	21.26	23.39	8.46	
Alsop.....	53	75.00	13.25	13.47	20.84	22.68	8.31	
Average.								
Average.								
Official.....	45.2	70.84	13.18	12.90	20.50	22.61	7.97	
Chrome added all at once.	44	69.70	13.33	12.95	20.44	22.73	7.87	
Chromed ½ hour.....	44.7	70.20	13.32	13.34	20.78	22.88	8.28	
Chromed 1 hour.....	44.6	69.33	13.68	13.14	20.78	22.80	8.18	
Unchromed .	50.5	74.24	13.01	13.54	21.05	23.04	8.38	

These results do not warrant changing the method so as to shorten the time of Chroming to the extent tried. There does not appear to be much difference whether the Chrome is added all at once or in two portions (one-half at the beginning and the rest at least six hours before finishing). Mr. Mosbaugh is of the opinion that the method as it is at present is best.

There is no material difference between the averages obtained by the two methods and the results obtained by the collaborators are closest when adding the Chrome all at once.

Amount of Hide Powder for Analysis.

Experiments using such amount of wet hide as represents the following amounts of dry hide: (1) 12 grs., (2) 11 grs., (3) 10 grs., (4) 9 grs.

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	Wt. Wet Hide. Grams.	Per ct. Water in Wet Hide.	Dry Hide. Gms.	Per Cent. Non-tannins. Chest-nut.	Hem-lock.	Oak.	Que-bracho.	
Reed.....	46	70.00	13.80	12.65	20.37	22.34	7.45	
Alsop.....	50	72.25	13.87	12.84	20.60	22.65	7.87	
Hildebrand..	50	71.00	14.50	13.81	20.93	23.36	8.80	
Mosbaugh...	40	65.00	14.00	13.00	20.60	22.48	7.96	Hide No. 1
Mosbaugh...	36.5	64.50	13.00	13.12	20.78	22.79	8.19	Hide No. 2
Reed.....	42	70.25	12.50	12.85	20.51	22.55	7.55	
Alsop.....	50	74.00	13.00	12.85	20.41	22.68	7.67	
Hildebrand..	50	75.00	12.50	13.52	20.58	23.34	8.25	
Mosbaugh...	36	65.00	12.60	13.57	21.09	23.69	8.71	Hide No. 1
Mosbaugh...	33.5	64.50	11.90	13.86	21.40	24.15	8.73	Hide No. 2
Reed.....	38	70.25	11.30	12.95	20.57	23.04	7.60	
Alsop.....	44	74.00	11.44	12.92	20.46	22.79	7.76	
Hildebrand..	46	75.00	11.50	13.52	20.77	23.38	8.27	
Mosbaugh...	33	65.00	11.50	14.43	22.00	24.59	9.20	Hide No. 1
Mosbaugh...	30.7	64.50	10.90	14.02	22.11	24.60	9.40	Hide No. 2
Reed.....	34.5	70.25	10.25	13.32	20.87	23.37	7.70	
Alsop.....	40	74.00	10.40	13.25	20.67	23.10	7.84	
Hildebrand..	42	75.00	10.50	13.57	20.84	23.35	8.40	
Mosbaugh...	30	65.00	10.50	15.03	22.99	25.15	9.88	Hide No. 1
Mosbaugh...	28	64.50	9.90	....	22.76	25.10	10.31	Hide No. 2
Reed.....	35	73.50	9.28	13.36	20.74	23.10	7.90	
Alsop.....	36	74.00	9.36	13.36	20.90	23.63	7.97	
Hildebrand..	38	75.00	9.50	13.72	20.88	23.52	8.77	
Mosbaugh...	27	65.00	9.40	15.21	23.33	25.69	10.20	Hide No. 1
Mosbaugh...	25	64.50	8.90	15.60	23.01	25.12	15.60	Hide No. 2

Mr. Mosbaugh's results are not comparable with the rest, so I have not made an average. In all probability his results are due to using a hide squeezed so as to contain an unusually low percentage of water. Mr. Mosbaugh states that in his opinion the amount of hide powder used should be increased above (12 to 13) grams. I do not agree with him. Mr. Reed suggests 11.5 to 12.5 as giving the most satisfactory results. Probably the method should be left as it is. I will refer to these results again under Table No. 3.

3. EFFECT OF GREATER OR LESS AMOUNT OF WATER IN HIDE

	Wet Hide. Gms.	Per ct. Water in Wet Hide.	Dry Hide. Gms.	Per Cent. Non-tannins. Chest-nut.	Hem-lock.	Oak.	Que-bracho.	
Reed.....	60	80.00	12.00	12.82	20.73	22.73	8.09	
Alsop.....	60	80.00	12.00	13.37	20.77	23.12	8.11	
Reed.....	52	77.00	11.90	12.80	20.62	22.37	7.63	
Alsop.....	50	74.00	13.00	12.85	20.41	22.68	7.67	
Mosbaugh...	..	75.00	13.20	12.55	20.50	22.63	8.04	Hide No. 1
Mosbaugh...	..	75.00	13.20	12.41	20.15	22.40	7.93	Hide No. 2
Mosbaugh...	..	72.00	13.00	12.48	20.19	22.48	8.12	Hide No. 2
Reed.....	37.5	68.50	11.80	12.88	20.47	22.72	7.70	
Alsop.....	40	67.50	13.00	13.10	21.07	22.98	7.90	
Mosbaugh...	46	68.00	14.70	12.71	20.38	22.64	8.02	Hide No. 1
Mosbaugh...	42	64.00	15.00	12.80	20.21	22.54	7.80	Hide No. 1
Mosbaugh...	42	64.00	15.00	12.69	20.39	22.30	8.00	Hide No. 2
Reed.....	34.5	65.50	11.90	12.92	20.54	22.68	7.76	

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I think that a study of Tables No. 2 and No. 3 will show that different amounts of water in hide have a marked influence on the results, and that the differences in results obtained by collaborators are due largely to this cause and not to the different amounts of dry hide used. Mr. Reed states that the less water in hide the greater the difficulty of obtaining clear non-tannins, and that from 70 to 75% of water appears to be advisable. I could not obtain clear non-tannins with hide squeezed to contain 67.5% water. Mr. Reed also states that it was utterly impossible to obtain satisfactory filtrates with hide containing 64% water, when using an amount equivalent to 9 grams of dry hide. It will be noticed that Mr. Mosbaugh obtains results with 75 and 72% water and 13.2 and 13. grams dry hide that are comparable with his results when using 15. grams of hide containing 64% water. Therefore I do not see the necessity for increasing the amount of hide to be used for analysis, but rather to prescribe limits for the water content. I think that Mr. Reed's suggestion, 75 to 70% water, is a good one and should be put in the method.

4. EFFECT OF SHAKING FIVE MINUTES AS COMPARED WITH TEN MINUTES.

Minutes.	Chestnut. 5	Chestnut. 10	Hemlock. 5	Hemlock. 10	Oak. 5	Oak. 10	Quebracho. 5	Quebracho. 10	
Reed	13.13	12.84	20.70	20.58	22.85	22.68	7.80	7.63	
Alsop	12.92	12.84	20.41	20.60	22.71	22.65	7.76	7.87	
Hildebrand	13.42	13.51	20.97	20.90	23.13	23.17	8.66	8.69	
Mosbaugh	13.95	13.30	21.36	20.71	23.01	22.72	8.11	7.88	Hide No. 1
Mosbaugh	13.65	13.10	20.83	20.49	22.92	22.59	8.16	7.93	Hide No. 2
Average	13.41	13.12	20.85	20.66	22.92	22.76	8.10	8.00	

These results do not seem to warrant shortening the time for shaking to five minutes.

5. COMPLETE ANALYSES OF EXTRACTS.

	Chestnut Extracts. Reed.	Hildebrand.	Alsop.	Mosbaugh.	Average.
Water	55.63	55.61	55.95	55.56	55.69
Total Solids	44.37	44.39	44.05	44.44	44.31
Soluble Solids	44.03	44.25	43.88	44.07	44.06
Reds	.34	.14	.17	.37	.25
Non-tannins	12.84	13.51	12.84	13.30	13.12
Tannins	31.19	30.74	31.04	30.77	30.94
Hemlock Extract.					
Water	46.44	46.43	46.66	46.20	46.43
Total Solids	53.56	53.57	53.34	53.80	53.57
Soluble Solids	51.24	51.50	51.08	51.31	51.28
Reds	2.32	2.07	2.26	2.49	2.29
Non-tannins	20.58	20.90	20.60	20.71	20.70
Tannins	30.66	30.60	30.48	30.60	30.58

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5. COMPLETE ANALYSIS OF EXTRACTS.—Continued.

	Chestnut Extracts.				
	Reed.	Hildebrand.	Alsop.	Mosbaugh.	Average.
Oak Extract.					
Water	48.10	47.44	48.17	47.93	47.91
Total Solids	51.90	52.56	51.83	52.07	52.09
Soluble Solids	51.26	51.53	51.05	50.68	51.13
Reds	.64	1.03	.78	1.39	.96
Non-tannins	22.60	23.17	22.65	22.72	22.79
Tannins	28.66	28.36	28.40	27.96	28.34
Quebracho Extract.					
Water	50.60	50.37	50.37	50.60	50.49
Total Solids	49.40	49.63	49.63	49.40	49.51
Soluble Solids	48.68	48.83	48.83	48.98	48.83
Reds	.72	.80	.80	.42	.68
Non-tannins	7.63	8.69	7.87	7.88	8.02
Tannins	41.05	40.14	40.96	41.10	40.81

6. DRY CHROMED HIDE POWDER.

Mr. Mosbaugh sends results with three different lots of dry chromed hide as follows:

	Non-tannins.			
	Quebracho.	Hemlock.	Oak.	Chestnut.
Lot No. 1	7.50	20.60	22.18	12.74
Lot No. 2	7.80	20.31	22.00	12.81
Lot No. 3	7.66	20.00	22.23	12.60

Following are some results with hide sent to Mr. Reed and myself by Mr. Mosbaugh. These results were obtained in the course of routine work and are not from the samples sent out.

Reed.

	Chestnut.	
	Wet.	Dry.
No. 1	11.83	11.49
" 2	12.98	12.61
" 3	11.58	10.88
" 4	11.17	10.68
Quebracho.		
" 1	9.05	8.81

12.5 grams Dry Chromed Hide used. Wet Chromed Hide repreented 12.38 grams dry.

Shaken 10 minutes.

Alsop.

	Chestnut.	
	Wet.	Dry.
No. 1	14.90	15.09
" 2	17.04	17.30
Quebracho,		
" 1	7.58	7.92
" 2	8.23	8.36
" 3	7.88	7.97
" 4	7.68	7.93

12 grams Dry Chromed Hide used. Wet Chromed equal 13.50 dry.

Shaken 10 minutes.

These results are encouraging and further experiments which are to be made when the general collaborative work is sent out will probably furnish a better basis from which to determine whether it is practical to use the dry powder. If it can be made so that different lots will give comparable results a distinct advance will be made.

The results obtained by this Committee involved a considerable amount of work and I think that they justify it. The results ob-

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tained in the complete analysis of extracts are the closest that I have seen, when the character of the extracts are taken into consideration. They show that it is not advisable to squeeze hide powder so that it contains less than 70% water, and that when so squeezed more hide is required to give satisfactory non-tannins. That, when the water content is within proper limits, the equivalent of 11.5 to 14. grams of dry hide does not make any material difference in the percentage of non-tannins, and that therefore the amount prescribed by the method would seem to be satisfactory, and if slightly varied from, which is sometimes unavoidable, will not seriously affect the results.

W. K. Alsop,
Chairman Committee.

The conclusions to be drawn from this report pointed so strongly in favor of a definite moisture content for wet chromed hide powder of from 70% to 75%, and to the needlessness of adding the chrome alum in two portions, that the Referee incorporated the recommendations in the general collaborate prospectus.

The Referee sent out the following general instructions under Study I:

STUDY I.

ANALYSIS OF EXTRACTS.

Solid Quebracho Extract. Dilution 6 grams per liter.
Liquid Hemlock Extract. Dilution 14 grams per liter.
Complete analysis of both samples.

(a) Extracts are to be dissolved in water at 80° C., made up to 900 cc., with water at the same temperature and allowed to stand over night. Solutions are then brought to a temperature between 20° and 25° C., the particular temperature used to be stated, and the analysis proceeded with as per Official method, all solutions being at the definite temperature when pipetted and care being observed in keeping at the definite temperature during filtration for soluble solids.

Evaporation and drying of residues to be conducted by a method similar in effect to that of the combined evaporator and dryer, a description of which is enclosed. The temperature must not be allowed to go below 98° C., nor above 100° C. The time of

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evaporation and drying to be 16 hours. The Referee will welcome a comparison of this method of evaporation and drying with any other method or methods.

The hide powder for non-tannin determinations is to be chromed by adding all the chrome alum at once instead of in two portions. The amount of water in the wet hide powder must fall between 70% and 75% and the amount of wet hide used must represent between 12 and 13 grams of dry hide per each 200 cc. of the tannin solution. After shaking for 10 minutes, squeeze immediately through linen, add 2 grams kaolin and filter through No. 590 S. & S. filter paper, collecting when clear and evaporating 100 cc. The weight is to be corrected for the water contained in the wet hide powder. State percentage of water in wet hide and amount of wet hide taken.

Non-tannin determinations to be made on both extracts using the dry chromed hide powder furnished, in the proportion of 12½ grams per 200 cc. of the tannin solutions. The hide powder and tannin solution are not to be allowed to stand in contact, but to be shaken immediately upon adding one to the other.

(b) Solutions of extracts to be made as in "(a)", allowed to stand over night and total solids and non-tannins determinations to be made on solution pipetted at room temperature. That portion of solution used for soluble solids filtration, to be brought to exactly 20° C., and filtration immediately proceeded with, not keeping the solution at a definite temperature, as in "(a)" but permitting the influence of the room temperature. Allow the soluble solids filtrate to return to room temperature before pipetting. State room temperature and temperature of filtrate at conclusion of filtration.

Method of evaporation and drying and percentage of water in wet hide powder, with statement of same, and proportion of dry hide powder to be the same as in "(a)".

(c) Determination of soluble solids by means of the Berkefeld filter candle. Temperature and method of drying to be same as in "(a)". Non-tannins determinations to be omitted.

NOTE.—The Referee is, at present, unable to furnish the filter candle, but will forward it with description as to use, as early as possible.

The directions for use of the filter candle, sent to collaborators, will occur later in the report.

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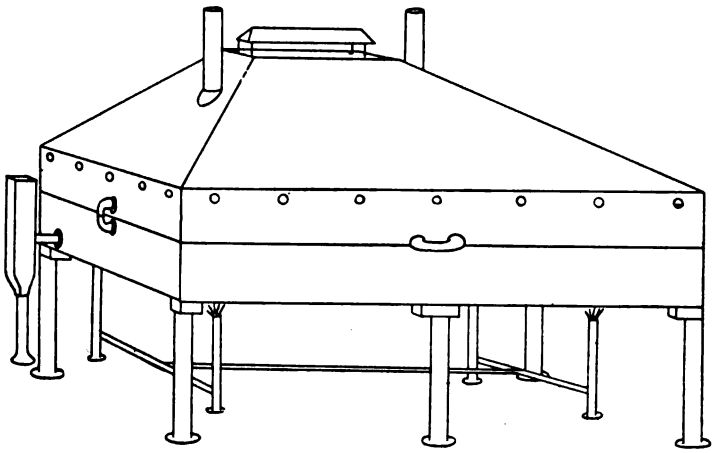
The drawing represents an oven that will hold 90 dishes, 2¾ inches in diameter. The water compartment, 31 inches long by 25 inches wide and 5 inches deep, is made of copper, the top, on which the dishes rest, should be made of heavy copper in order to insure a level surface, it is provided with the usual arrangement to insure a constant water level. The hood, which rests on the water-bath, is jacketed, with one inch space between the walls. These are perpendicular for 4 inches, and the distance between the cover of the water compartment and the top of the hood is 12 inches. It is fastened by hinges to the back of the water-bath, and is lifted by the handle shown. The holes shown in the drawing are 3 inches above the bottom of the hood, and ½ inch in diameter. They create a draught that carries off the steam, during evaporation, through the opening of the hood, which is 1 inch wide and 8 inches long.



A Combined Evaporator and Dryer.

The hood is heated by steam from the boiling water, which passes through pipes at each end of the oven, and escapes through the openings in the top. These pipes, 1 inch in diameter, are elbows that fit together when the hood is down.

The whole apparatus rests on an iron stand, and is heated by means of four burners.

Under Study I "(a)" and "(b)", the Referee submitted samples of Solid Quebracho Extract and Liquid Hemlock Extract to twenty-five analysts and received replies from twenty-two.

The analyses are submitted below in tabulated form:

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TABLE I.—STUDY I (a) QUEBRACHO EXTRACT.

ANALYST.	Total Solids.	Soluble Solids.	Insolubles.	Non-tannins.	Tannins.	Non-tannins. Dry Chromed Hide.	Temperature Degree C.	Wet Hide. Grams.	Moisture. Per cent.	Dry Hide. Grams.	Drying Method.	
W. H. Teas	89.85	82.29	7.56	12.52	69.77	16.45[2]	22	50	70.00	15.00	E. & D.	
H. T. Wilson.....	90.10	82.54	7.56	11.54	71.00	11.96	24	40	65.24	13.90	W. O.	
J. V. R. Evans	89.91	82.55	7.36	12.52	70.03	16.45[2]	21	50	70.00	15.00	E. & D.	
W. K. Alsop and A. W. Hoppenstedt	89.21	82.21	7.00	11.46	70.75	18.50[2]	23	50	73.50	13.25	E. & D.	
F. W. Ackermann.	89.33	82.33	7.00	11.90	70.43	12.17	...	50	75.50	12.75	E. & D.	
Chas. Eachus	88.33	81.77	6.56	10.59	71.18	17.70[2]	...	48.5	74.91	12.10	S. O.	Tempt. Analysis, 28°.
F. P. Veitch[1].....	50.62	80.70	9.92	11.35	69.35	10.91	...	50	78.00	11.00	W. O.	Tempt. T. S., 30° ; S. S., 20°; N. T., 28°–30°.
H. H. Hurt	88.74	80.26	8.48	11.26	69.00	....	...	50	76.40	11.80	W. O.	Tempt. T. S., 30° ; S. S., 20°;
M. F. Nichols.....	88.66	81.50	7.16	11.79	69.71	11.33	24	45	74.00	11.70	E. & D.	N. T., 28°–30°.
E. G. Wilmer	88.73	81.57	7.16	13.45	68.12	12.56	24.5	44	72.79	11.97	S O.	Tempt. drying, 93°–96°.
F. W. Alden	89.22	82.08	7.14	11.67	70.41	11.28	24.5	49	76.11	11.71	S. O.	Tempt. drying, 93°–96°.
G. D. McLaughlin	91.98	83.59	8.39	11.77	71.82	12.38	24	42	70.45	12.41	W. O.	
C. R. Delaney.....	88.76	80.83	7.93	12.49	68.34	12.75	24.5	35	67.84	11.26	W. O.	
G. A. Kerr.....	90.93	84 91	6.02	13.17	71.74	20.49[2]	25	60	78.00	13.20	W. O.	Time, 3 hours.
J. R. Mardick[1]	90.32	85.33	4.99	15.39	69.94	....	22	41.8	71.28	12.00	A. B.	Tempt. drying, 100°–103° ,
T. J. Mosser	88.64	80.10	8.54	12.66	67.44	19.34	24	50	80.00	10.00	S. O.	Tempt. drying, 96°.
F. H. Small.....	88 46	80.58	7.88	10.97	69.61	12.80[2]	25	43	71.00	12.50	O. O.	
H. C. Reed	89.16	81.07	8.09	11.21	69.86	11.73	22	48	73.64	12.67	E. & D.	
C. W. Norris.....	89.06	81.01	8.05	11.07	69.94	12.72[2]	25	45	70.65	13.21	E. & D.	
F. R. Mosbaugh ..	89.50	81.66	7.84	11.81	69.85	11.83	22.5	40	68.80	12.48		
W. A. Fox	88.83	80.50	8.33	11.06	69.44	20.17[2]	21.5	50	74.00	13.00	E. & D.	
Average	89.33	81.75	7.58	11.83	69.92	14 40	23.5	46.8	72.78	12.63		
Maximum	91.98	83.59	8.54	13.45	71.82	20.49	25	60	80.00	15.00		
Minimum	88.33	80.10	6.02	10.59	67.44	10.91	21	35	65.24	10.00		
Greatest difference	3.65	3.49	2.52	2.86	4.38	9.58	4	25	14.76	5.00		

[1] Omitted from average for probable error. [2] Not detannized or highly colored. E. & D.—Evaporator and Dryer. W. O.—Water Oven. S. O.—Steam Oven. A. B.—Air Bath. O. O.—Oil Oven.

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TABLE II.—STUDY I (b) QUEBRACHO EXTRACT.

ANALYST.	Total Solids.	Soluble Solids.	Insolubles.	Non-tannins.	Tannins.	Room Temperature. Degree C.	Wet Hide. Grams.	Moisture. Per cent.	Dry Hide. Grams.	
W. H. Teas	89 85	82.23	7.62	12.51	69.72	25	50	70.00	15.00	
H. T. Wilson....	90.35	83.25	7.10	11.18	72.07	26 –28	41	66.96	13.55	
J. V. R. Evans ..	89.95	82.23	7.72	12.51	69.97	27	50	70.00	15.00	
W. K. Alsop and A.W.Hoppenstedt	89.00	83.08	5.92	11.50	71.58	30	50	73.50	13.25	
F.W. Ackermann	89 50	82.83	6.67	12.19	70.64	29 5	50	75.50	12.75	
Chas. Eachus ...	88.50	81.59	6.91	10.71	70.88	28	48 5	74.91	12.10	
F. P. Veitch	90.62	81.66	8 96	11.35	70.31	.....	50	78.00	11.00	Tempt. T. S , 30° ; S. S., 20° ; N. T., 28°–30°.
H. H. Hurt	88.74	81.42	7.32	11.26	70 16	.....	50	76.40	11.80	Tempt. T. S., 30° ; S. S., 20° ; N. T., 28°–30°.
M. F. Nichols...	88.83	81.00	7.83	11.79	69.21	30—28	44	72.00	12 32	
E. G. Wilmer[1] ..	89 30	79.59	9.71	12.19	67.40	27.5	46	73.68	12.00	
F. W. Alden	89.07	82.62	6.45	12.00	70.62	26	49	76 11	11.71	
G.D McLaughlin	90.47	84.25	6.22	11.54	72.71	29	42	70.45	12.41	
C. R. Delaney...	89.67	83.96	5.71	13.13	70.83	30	40	68.07	12.77	
G. A. Kerr[1]	90.27	85.70	4 57	12.92	72.78	22	50	65.00	17.50	
T. J. Mosser.....	87.50	79 66	7.84	12.67	66.99	26	50	80.00	10.00	
F. H. Small.....	87.91	80.27	7.64	10.96	69 31	27	43	71.00	12.50	
H. C. Reed	89.48	82.05	7.43	11.23	70 82	25	48	73.64	12.67	
C. W. Norris....	88.97	82.61	6.33	10.98	71.66	29	45	70.65	13.21	
F. R. Mosbaugh	89.45	81.49	7.96	11.77	69.72	23	40	68.80	12.48	
W. A. Fox	88.75	80.67	8.08	10 76	69.91	22.5	50	74.00	13.00	
Average	89.26	82.05	7.21	11.67	70.38	27	6.67	72.77	12.64	
Maximum	90.62	83.96	8.96	13.13	72.71	30	50	80.00	15.00	
Minimum	87.50	80.27	5.71	10.71	66.99	22.5	40	66.96	10.00	
Greatest Dif	3.12	3.69	3.25	2.42	5.72	7.5	10	13.04	5.00	

[1] Omitted from average for probable error.

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TABLE III.—STUDY I (a) HEMLOCK EXTRACT.

ANALYST.	Total Solids.	Soluble Solids.	Insolubles	Non-tannins.	Tannins.	Non-tannins. Dry Chromed Hide.	Temperature. Degree C.	Wet Hide Grams.	Moisture. Per cent.	Dry Hide. Grams.	Drying Methods.	
W. H. Teas.....	50.09	47.29	2.80	20.68	26.61	23.20[2]	22	50	70.00	15.00	E. & D.	
H. T. Wilson	50.34	46.73	3.61	20.42	26.31	21.12[2]	24	40	65.24	13.90	W. O.	
J. V. R. Evans ...	49.91	47.40	2.51	20.91	26.49	26.73[2]	21	50	70.00	15.00	E. & D.	
W. K. Alsop and A. W. Hoppenstedt	49.74	46.97	2.77	19.76	27.21	25.49[2]	23	50	73.50	13.25	E. & D.	
F. W. Ackermann.	50.08	47.20	2.88	20.54	26.66	21.76	23.5	50	73.50	13.25	E. & D.	
Chas. Eachus	49.71	47.01	2.70	19.51	27.50	25.56[2]	...	48.5	74.91	12.17	S. O.	Tempt. of Analysis, 28°.
F. P. Veitch[1].....	49.70	44.98	4.72	19.68	25.30	21.04	...	50	78.00	11.00	W. O.	Tempt. T. S., 30° ; S. S., 20° ; N. T., 28°–30°.
H. H. Hurt[1]	49.75	44.97	4.78	20.21	24.76	...	..	50	76 40	11.80	W. O.	Tempt. T. S., 30° ; S. S., 20° ;
M. F. Nichols.....	49.50	46.93	2.57	19.80	27.13	20.64	24	45	74.00	11.70	E. & D.	N. T., 28°–30°.
E. G. Wilmer.....	49.90	47.62	2.28	20.13	27.49	22.62	24.5	44	72.79	11.97	S. O.	Tempt. drying 93°–96°.
F. W. Alden	50.00	47.11	2.89	19.45	27.66	22.89	24.5	49	76.11	11.71	S. O.	Tempt. drying 93°–96°.
G. D. McLaughlin	50.15	46.56	3.59	20.55	26.01	21.78	23	42	71.37	12.03	W. O.	
C. R. Delaney.....	50.10	46.71	3.39	21.52	25.19	22.54	24.5	35	67.84	11.26	W. O.	
G. A. Kerr[1].....	50 81	47.82	2.98	22.33	25.49	25.35[2]	25	40	65.00	14.00	W. O.	Time, 3 hours.
J. R. Mardick[1]	49.39	46.67	2.72	17.68	28.99	....	22	41.8	71.28	12.01	A. B.	Tempt. drying 100°–103°.
T. J. Mosser	50.56	47.31	3.25	20.82	26.49	25 04	24	50	80.00	10.00	S. O.	Tempt. drying 96°.
F. H. Small.....	50.87	47.95	2.92	20.23	27.72	23.55[2]	25	43	71.00	12 50	O. O.	
H. C. Reed.....	50.02	47.10	2 92	19.50	27.60	22.46[2]	22	48	73.64	12.65	E. & D.	
C. W. Norris[1]	49.69	45.25	4.44	20.49	24.76	23.49[2]	25	45	70 65	13.21	E. & D.	
F. R. Mosbaugh...	50.07	47.22	2.85	20 48	26.74	20.71	22 5	40	68.80	12.48		N. T. with unofficial D. C. H.
W. A. Fox.....	50.00	45.97	4.03	19.24	26.73	26.44[2]	21.5	50	74.00	13.00	E. & D.	
Average	50.07	47.07	3.00	20.23	26.84	23.35	23.3	45.9	72.30	12.62		
Maximum	50.87	47.95	4.03	21.52	27.72	26.73	24.5	50	80.00	15.00		
Minimum.....	49.50	45.97	2.28	19.24	25.19	20.64	21	35	65.24	10.00		
Greatest difference	1.37	1.98	1.75	2.28	2.53	6.09	3.5	.5	4.76	5.00		

[1] Omitted from average for probable error. [2] Not detannized or highly colored. E. & D.—Evaporator and Dryer. W. O.—Water Oven. S. O.—Steam Oven. A. B.—Air Bath. O. O.—Oil Oven.

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TABLE IV.—STUDY I (b) HEMLOCK EXTRACT.

ANALYST.	Total Solids.	Soluble Solids.	Insolubles.	Non-tannins.	Tannins.	Room Temperature. Degree C.	Wet Hide. Grams.	Hide Powder. Moisture. Per cent.	Dry Hide, Grams.	
W. H. Teas	49.85	47.40	2.45	20.77	26.63	25	50	70.00	15.00	
H. T. Wilson....	50.33	47.41	2.92	20.66	26.75	26—28	41	66.96	13.55	
J. V. R. Evans..	49.67	47.62	2.05	20.75	26.87	27	50	70.00	15.00	
W. K. Alsop and A.W.Hoppenstedt	50.00	47.67	2.33	19.87	27.80	30	50	73.50	13.25	
F.W. Ackermann	50.20	47.97	2.33	20.37	27.60	29	50	73.50	13.25	
Chas. Eachus ...	49.79	47.07	2.72	19.55	27.52	28	48.5	74.91	12.17	
F. P. Veitch	49.70	47.10	2.60	19.68	27.42	.....	50	78.00	11.00	Tempt. T. S., 30° ; S. S., 20°–30° ; N. T., 28°–30°.
H. H. Hurt	49.75	47.07	2.68	20.21	26.86	... ..	50	76.40	11.80	Temp. T. S., 30° ; S. S., 20°–30° ; N. T., 28°–30°.
M. F. Nichols...	49.71	46.78	2.93	20.46	26.32	30—28	44	72.00	12.32	
E. G. Wilmer...	50.07	46.89	3.18	20.74	26.15	27.5	46	73.68	12.11	
F. W. Alden	49.83	46.69	3.14	19.45	27.24	26	49	76.11	11.71	
G.D.McLaughlin	49.84	46.94	2.90	20.17	26.77	32	42	71.37	12.03	
C. R. Delaney...	49.94	47.68	2.26	21.82	25.86	30	40	68.20	12.72	N. T. 10.2 gm. Dry Hide = 22.04.
G. A. Kerr.....	50.47	47.72	2.75	21.08	26.64	22	50	65.00	17.50	
T. J. Mosser	50.56	47.69	2.87	21.22	26.47	26	50	80.00	10.00	
F. H. Small.....	50.88	48.02	2.86	20.12	27.90	27	43	71.00	12.47	
H. C. Reed	49.87	47.53	2.34	19.54	27.99	25	48	73.64	12.65	
C. W. Norris....	49.76	46.59	3.17	20.09	26.50	29	45	70.65	13.21	
F. R. Mosbaugh.	50.11	47.11	3.00	20.35	26.76	23	40	68.80	12.48	
W. A. Fox.....	49.98	46.79	3.19	19.16	27.63	25	50	74.00	13.00	
Average.....	50.02	47 29	2.73	20.30	26.99	27.1	46.8	72.39	12.86	
Maximum	50.88	48.02	3.19	21.82	27 99	32	50	80.00	17.50	
Minimum.....	49.67	46.59	2.05	19.16	25.86	22	40	65.00	10.00	
Greatest dif.....	1.21	1.43	1.14	2.66	2.13	10	10	15.00	7.50	

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COLLABORATORS' NOTES AND METHODS RELATIVE TO STUDY I "(a)" AND "(b)".

W. K. Alsop and A. W. Hoppenstedt.

"The filtrations for soluble solids by method "(a)" were very slow. By method "(b)" the soluble solids filtrates were at room temperature when filtrations were finished. The dry chromed hide powder did not work at all well, the non-tannin filtrates being colored, filtering slowly and requiring to be returned through the filter a number of times in order to get a clear filtrate.

The non-tannin filtrates from the solutions at 23° were more colored than the others.

These samples were not analyzed until August 24th, which may partially account for the failure of the dry chromed hide to give satisfactory results.

Results obtained in this laboratory with the dry chromed hide on another set of samples were much more satisfactory. These were analyzed at once upon receipt of samples. It is our opinion that this hide has deteriorated in quality since it was received. That it has dried out and become hard, and for that reason will not properly detannize the solutions with the treatment given above. Dry chromed hide has been made in this laboratory that was satisfactory when first made, but in a short time was worthless.

Yocum-Eachus Laboratory.

"The soluble solids portions which were cooled to 20° C., (method "(b)") rose to the temperature of the laboratory by the time there was enough of the filtrate through. It took four hours to make the soluble solids filtration. The dry chromed hide powder gave dark colored filtrates and high non-tannins."

F. P. Veitch and H. H. Hurt.

"We have not followed the instructions in all cases. All dryings were for 16 hours, and were made on the bottom shelf of the ordinary water oven, and all filtering and pipetting were done at the laboratory temperature (28° to 30° C.), unless otherwise stated in the tables. Specific gravity of the spent liquors was not determined, and in calculating percentages cubic centimeters are used as grams."

F. W. Alden and E. G. Wilmer.

"All dryings were made in the following manner: Aluminum

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pans (3⅜ inches by 1¼ inches) containing the liquor to be evaporated were placed on a water-bath over direct steam until dry, which occupied about 1½ hours. They were then placed in a dessicator until evening, when they were removed to a steam-heated oven with a temperature between 93° and 96° C., and there remained over night and weighed the next morning. Each dish was cooled in a dessicator by itself and weighed as quickly as possible."

C. R. Delaney.

"You will notice the relatively large number of analyses both of the Solid Quebracho Extract and of the Liquid Hemlock Extract. The reason for this is, that I could not get results that were concordant with comparatively small changes in the temperature; in fact, I found that solutions made at different times, notwithstanding that the greatest care was used, gave different results when they were finished. All my dryings were made in a large water oven capable of running for 30 hours without being refilled, at a temperature from 98° to 100° C., and all dryings were as far as possible uniform. Much as I would have liked to have kept the temperatures uniform, I found that it was impossible to do so as the fluctuations of temperature for the last month have been great, some days being down to 20° C., and a couple days after over 30°.

In both the Quebracho and Hemlock analyses I have made two other analyses with the dry chromed hide powder which you furnished, but the results were so very far apart that I did not think it would be advisable to include them in any table of averages."

These results are not included in this report as they are merely confirmatory of our previous knowledge of the effect of temperature on tanning solutions.

G. A. Kerr.

"We think the time for drying residues, 16 hours, is too long in an oven through which there is a rapid circulation of air.

The dilutions in both cases were allowed to cool over night, and the temperature reduced from room temperature (22° C.) to 20° C. Upon completion of filtration of soluble solids, solution was found to have returned to room temperature, which was constant at 22° C., (exceptionally cool for Central Virginia, in August).

In the analysis as per Study I (a) the quantity of hide powder used seemed insufficient, we, therefore, increased it in this case to

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an equivalent of 17.5 grams, dry, with the result of raising the tannin figure considerably.

I think the ranges of temperature to which our dilute liquors were subjected were too small to indicate anything relative to soluble solids. Our results show lower insolubles at 20° than at 25°; this would indicate, the error caused by irregularities in the quality and character of the filter paper, was much greater than that caused by 5° C., in temperature."

J. R. Mardick.

"Study I (a) were analyzed both by the official method and by our own method. As you know, we aim to follow as near to tannery conditions as possible, for this reason we believe that the solutions ought to be acidified with some organic acid (to .4-.5%) before analysis. We are inclined to think that the quantity recommended for analysis is rather weak. It ought to be at least two to three times stronger, as the same amount of hide powder (12 grams) will absorb completely all the tannins. Hope that Berkefeld filter candle will enable us to use stronger liquors, although the same end can be accomplished by acidifying the solutions and thus settling the reds."

Mr. Mardick remarks that the dry hide powder furnish contains soluble sulphates, hence not used. His analyses according to the tanner's method and by the method of acidification are here submitted:

STUDY I (a)—EXTRACTS—ACCORDING TO TANNER'S METHOD.

	Total Solids. Per cent.	Soluble Solids. Per cent.	Non-Tannins. Per cent.	Tannins. Per cent.	Insolubles. Per cent.	Weight taken for Analysis.	Temperature of Analysis.
Quebracho.....	90.32	78.00	11.69	66.39	12.32	16. g.	22° C.
Hemlock	49.39	43.84	19.61	24.23	5.85	28.56	22° C.

Remarks: Tanner's Method. The details of this method are given in Journal Society Chemical Industry, Dec. 31, 1904, or Shoe and Leather Reporter, Jan. 26 and Feb. 2, 1905.

STUDY I (a)—EXTRACTS—THE SOLUTION ACIDIFIED TO 0.50%, (MIXTURE 2/3 ACETIC AND 1/3 LACTIC).

	Tannins. Per cent.	Temperature of Analysis.
Quebracho.....	64.75	22° C.
Hemlock	21.40	22° C.

Remarks: Acidiified Analysis Solution. This method brings the conditions on tannery basis, accelerates the filtration just about five times over the official method, and clarifies the solution by settling the reds.

NOTE.—Method of Evaporation, Drying, &c., same as in Tables 1 and 3.

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F. R. Mosbaugh.

"Note that there is very little difference in results of Study I (a) and (b), which is probably accounted for by the fact, that in (b) it was only necessary to cool solution about 2½°, as maintained laboratory temperature of 22-23° C., throughout. In my opinion, the temperautre factor should certainly be considered, and think 20-25° C., the most desirable."

C. W. Norris.

Mr. Norris uses the combined evaporator and dryer and has the following to say concerning it:

"In the construction of my oven I supply the heat with a copper coil (which has a number of fine perforations for the live steam to enter). The steam causes *no* bumping.

The temperature upon the drying plate is 90° C., during the evaporation (when the ventilator is open).

The temperature is 99.5° C., during the drying.

The evaporation of 100 cc. takes from 4½ to 5 hours."

SOLUBLE SOLIDS AND INSOLUBLES.

The two extracts submitted, offered as severe a test upon the method of filtration as could be presented.

As for uniformity, in the case of the Quebracho Extract there is but little choice between the two methods, while with the Hemlock Extract the uniformity is somewhat in favor of Method B. However, the advantage gained from this method is hardly sufficiently marked to warrant its adoption in lieu of the provisional method which we now have, limiting the temperature of filtration within the range of 20° to 25° C. It is true that the two extract solutions were of so slow filtration that room temperature was attained practically in every instance by the time the filtrations were concluded, and it is evident from the tables that the insolubles did go back into solution to some little extent with the rise of temperature. With a rapid filtering solution, a re-solution of insolubles would not obtain to such an extent, but the Referee had in mind the embracing of all possibilities in submitting extracts of the character he did.

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Since putting out the collaborative work, the Referee has done some little experimenting with the so-called time contact method suggested by Mr. Small. Results would seem to indicate that by judicious use of this method, improvements can be shown over any of the methods hitherto suggested. The Referee would therefore recommend that especial attention be given to this method in next year's work. Briefly stated, the modus operandi, is as follows: Solution to be filtered is stirred with 1 gram of kaolin, thrown upon filter paper and the filtrate returned to the filter, thus keeping filter full, for one hour. Solution on filter is then removed either by decanting or pipetting and replaced with fresh unfiltered solution, collecting the 100 cc. for evaporation after discarding the first 10 cc. of filtrate, or provided filtrate is not clear, the first 100 cc., coming clear after discarding the 10 cc. It might be imagined that the decanting or pipetting of the solution from the filter would be liable to produce cloudy filtrates, but such does not seem to be the case, the kaolin apparently forming a quite compact mat upon the filter. It is suggested that the determination of soluble solids using this method, be conducted as follows: Solution is made up to mark in morning at room temperature, thoroughly stirred with 1 gram of kaolin, thrown upon paper and filtrate returned for one hour. At expiration of this time have portion of solution cooled to 20° C., and proceed with filtration of cooled solution after removing the solution from the filter.

By Method B, the cooled solution is gradually returning to room temperature during the filtration of 250 cc., while with the proposed method, it is returning to room temperature during the filtration of 100 cc. or for a much shorter time. The Referee is confident that this method will show a more just comparison between slow filtering and rapid filtering solutions, than is now given by the official method, since the time of a slow filtration will be lessened and of a rapid filtration, increased.

The labor entailed may be somewhat greater with rapid filtering solutions, but is more than offset by the labor and time saved in case of slow filtrations.

The Referee presents below comparisons of the time contact method with the Official method. The results were obtained dur ing regular course of work.

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TABLE V.—TABLE SHOWING COMPARATIVE RESULTS OF TIME CONTACT METHODOF FILTRATION WITH OFFICIAL METHOD.

EXTRACT	Quebracho.		Quebracho.		Quebracho.		Quebracho.		Quebracho.		Quebracho Solid.		[1]Quebracho Solid.		Quebracho.	
Method.	T. C.	O.	T. C.	O.	T. C.	O.	T. C.	O.	T C.	O.	T. C.	O.	T. C.	O.	T. C.	O.
Total Solids ..	48.01	48.01	49.20	49.20	49.33	49.33	49.43	49.43	49.42	49 42	82.26	82.26	82.12	82.12	48.13	48.13
Soluble Solids	46.30	46.24	47.80	47.76	47.60	47.51	47.85	47.74	47.91	47.81	79.81	79.91	81.57	81.21	46.87	46.72
Insolubles	1.71	1.77	1.40	1.44	1.73	1.82	1.58	1.69	1.51	1.61	2.45	2.35	.55	.91	1.26	1.41

TABLE V.—(CONTINUED).

EXTRACT.	[1]Chestnut.		[1]Chestnut.		[1]Chestnut.		[1]Chestnut.		[1]Chestnut.		[1]Chestnut.		Quebracho.		Quebracho.	
Method.	T. C.	O.	T. C.	O.	T. C.	O.	T. C.	O.	T. C.	O.	T. C.	O.	T. C.	O.	T. C.	O.
Total Solids ..	41.34	41.34	39.68	39.68	38.66	38.66	40.01	40.01	40.30	40.30	40.12	40.12	47.11	47.11	47.92	47.92
Soluble Solids	40.85	40.84	39.21	39.28	38.35	38.40	39.66	39.70	39.11	39.14	39.64	39.63	45.30	45.22	46.16	45.98
Insolubles	.49	.50	.47	.40	.31	.26	.35	.31	1.19	1.16	.48	.49	1.81	1.89	1.76	1.94

[1] Time for filtration shorter by official than by time contact Method.

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The Referee would have liked to have been able to show results from a greater variety of extracts, but time did not permit.

The immediate cooling of solutions, as shown by the report of the Chairman on Soluble Solids, seems to offer no solution of the problem, as the insolubles appear to return to solution during filtration, as in Method B, owing to rise in temperature.

The report of the Committee apparently shows no advantage to be gained by reducing the amount of kaolin, and filter paper No. 590, S. & S., is apparently more uniform than the Swedish No. 1 F, and no change should be made here.

A rather interesting feature of the Soluble Solids Committee report is found in the tests which were made to show that reds once precipitated in a strong solution are not immediately redissolved upon dilution with cold water.

The results are sufficiently commented upon by the Chairman of the Committee in his remarks relative to the report.

The filtration with asbestos mat gave entirely negative results.

Other methods are sufficiently commented upon in the report by the Chairman, with the exception of the Berkefeld filter candle filtration, which, owing to its adoption by the International Association of Leather Trades Chemists, was thought best to be given a place in the work this year. The report of Mr. Wilson's committee on the filter candle filtration was not sufficiently conclusive one way or the other and the Referee considered it wise to obtain the filters directly from Dr. Parker, the originator and strong advocate of this method of filtration. With the candles was sent a detailed description as to their preparation and method of use, which is here given.

Study I (c).

Determination of Soluble Solids by means of the Berkefeld Filter Candle.

Before use the candle is to be prepared as follows: Remove thistle tube and rubber ring from candle and place the candle in a 10% solution of pure hydrochloric acid. Place over water-bath or air oven and keep warm for 12 hours. Pour off hydrochloric acid and fit up candle, washing with distilled water until free from iron. A description of the method of fitting up the filter candle is herewith enclosed.

I. Comparative soluble solids of the sample of solid Quebracho

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Extract already furnished and fresh sample of Hemlock Extract using S. & S. No. 590, single-pleated filter paper as per Official method and the Berkefeld filter candle as per enclosed directions. Filtrations to be conducted between 20 and 25° C., and the temperature stated, care being observed that the temperature of the solutions by filter and candle filtration is the same.

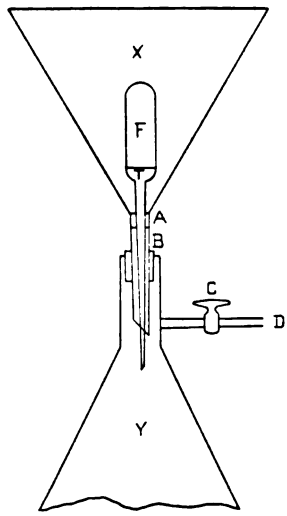
II. Results on soluble solids with the Quebracho and Hemlock Extracts, using the Berkefeld filter only, by evaporation of the 1st, 2nd, and 3rd 100 cubic centimeters passing through the filter (and as many more as is convenient), after discarding the 1st 150 cc., in order to prove whether there is a tannin absorption or not.

NOTE.—The Referee will welcome any results the collaborators may be willing to offer in addition to the work outlined.

No non-tannins will be required.

TOTAL SOLIDS MUST BE GIVEN.

State the time of filtration in every instance when paper is used as well as with the filter candle.



The stem of the funnel X is passed through a cork B fitting into the neck of the receiving flask Y. Capacity of Y about 500 cc.

The receiving flask Y is connected to a vacuum pump through tube D fitted with stop-cock C. A vacuum gauge is connected be-

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tween pump and cock C. The filter candle F is connected with the receiving flask Y by thistle tube T, stem of which passes through a cork A in stem of funnel X.

The operation is as follows: The solution to be filtered, is poured into funnel X, cock C opened and the vacuum pump run until a vacuum of 40 mm., is obtained. Cock C is then shut and 150 cc., allowed to filter. Vacuum is broken and the 150 cc. discarded. Vacuum is now renewed to 40 mm. and 100 cc., collected for evaporation. For each successive 100 cc., the vacuum must be broken, flask emptied and vacuum renewed to 40 mm.

If desired a separatory funnel of about 500 cc. capacity, can be used for the receiving flask Y.

If the candle becomes clogged it may be brushed with a toothbrush during operation.

The results from the various analysts following the above instructions are tabulated below.

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TABLE VI.—STUDY I (c) BERKEFELD CANDLE—QUEBRACHO EXTRACT.

ANALYST.	Total Solids.	Soluble Solids. Official. F. P No. 590.	Ins.	Time. Min.	Soluble Solids. Berkefeld. 1st 100 c.c.	Ins.	Time. Min.	Vac. M.M.	Soluble Solids. Berkefeld. 2nd 100 c.c.	Ins.	Time. Min.	Vac. M.M.	Soluble Solids. Berkefeld. 3rd 100 c.c.	Ins.	Time. Min.	Vac. M.M.	
W. H. Teas	90.00	82.77	7.23	...	86.78	3.22	20	40	85.70	4.30	29	40	85.45	4.55	64	40	
E. G. Wilmer	88.10	....	...	...	77.25	10.85	30	40	77.87	10.23	60	40	76.85	11.25	95	40	
J. R. Mardick	90.15	83 78	6 37	27	83.28	6.87	25	40	82.15	8.00	44	40	80.92	9.23	57	40	
F. P. Veitch	90.40	81.76	8.64	120	84.15	6 25	2	40	84.86	5.54	15	40	83.56	6.84	50	40	
J. V. R. Evans	90.25	81.95	8.30	360	86.35	3.90	15	40	86.83	3.42	20	40	86.83	3 42	25	40	
F. H. Small	88.04	80.58	7.46	265	87.59	.45	30	40	....	....	..	...	....	....	..	...	
F. H. Small	88.33	....	...	...	87.06	1.27	15	152	86.74	1.59	25	152	86.06	2.27	30	152	
F W. Alden	87.27	....	...	...	78.10	9.17	25	110	77.84	9 43	25	40	77.36	9.91	40	40	
C. W. Norris	88.14	80.78	7.36	180	87.72	10.42	14	40	87.79	10.35	17	40	87.61	10.53	20	40	
H. T. Wilson	89.95	82.21	7.74	145	81.78	8.17	26	152	81.41	8.54	26	152	82.04	7.91	26	152	4th Filt., S. S., 81.99, candle used. 5th Filt., S. S., 82.56, not official.
A. W. Hoppenstedt	88.79	81.17	7 62	306	86.00	2.79	14	400	84.17	4.62	..	400	82.83	5.96	..	400	
H. C. Reed	87.87	80.03	7.84	240	83.45	4.42	11	152	82.43	5.44	10	152	81.03	6.84	18	152	4th Filt., S. S., 80.83. Candle used, not official.
G. D. McLaughlin.	88.74	81.70	7.04	120	81.71	7.03	14	50	81.91	6.83	7	100	82.34	6.40	12	100	Candle, used, not official.

Note: Temperature omitted on account of wide variations in results.

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TABLE VII.—STUDY I (c) BERKEFELD FILTER CANDLE—HEMLOCK EXTRACT.

ANALYST.	Total Solids.	Soluble Solids. Official. S. S. Per ct.	Ins.	Time. Min.	Soluble Solids. Berkefeld. 1st 100 c.c.	Ins.	Time. Min.	Vac. M.M.	Soluble Solids. Berkefeld. 2nd 100 c.c.	Ins.	Time Min.	Vac. M.M.	Soluble Solids. Berkefeld. 3rd 100 c.c.	Ins.	Time Min.	Vac. M.M.	
W. H. Teas	50.24	46.75	3.49	....	48.94	1.30	15	40	49.18	.96	21	40	48.93	1.29	40	40	
E. G. Wilmer....	50.13	47.59	2.54	160	44.20	5.93	210	40	40.68	9.45	720	40	41 83	8.30	900	40	
J. R. Mardick....	49.20	46.51	2.69	62	45.74	3.46	98	40	45.47	3.73	166	40	45 25	3.96	270	40	
F. P. Veitch	49.99	47.02	2.97	240	41.23	8.76	300	40	43.97	6 02	360	40	....	....	...	...	
J. V. R. Evans...	50.28	47.21	3.07	420	48.38	1.90	15	40	47.99	2.29	20	49	47.92	2.36	25	40	
F. H. Small.....	51 36	47.27	4.09	270	46.70	4.66	330	40	....	...	...	...	....	....	...	...	
F. H. Small.....	51.44	....	...	....	48.06	3.38	85	152	46.05	5 39	285	152	....	....	...	...	
F. W. Alden	50.01	45 14	4 87	1140[1]	....	....	...	...	....	...	...	...	....	....	...	...	[1] Approximate.
F. W. Alden	49 82	....	...	....	35.57	14.25	...	40	....	...	...	...	....	....	...	...	
F. W. Alden	49 93	....	...	....	43.57	6.36	278	60	43.61	6.32	180	50	38.14	11.79	Over night	40	
C. W. Norris.....	50.33	46.38	3.95	240	45.56	4.77	300	40	45.31	5.02	320	40	44.87	5.46	370	40	
M. F. Nichols....	50.64	45.21	5.43	480	48.57	2.07	42	..	46.14	4.50	330	...	46.00	4.64	420	...	Vac. pump left on throughout, running full capacity.
H. T. Wilson	50 48	16.06	4.42	360	45.59	4.89	30	152	45.88	4 60	22	152	45.96	4.52	10	152	4th & 5th Filt. cloudy, candle used. Paper filt. Sli. cloudy, not official.
A. W. Hoppenstedt	50.62	46.41	4.21	600	46.88	3 74	59	400	47.09	3.53	...	400	47.45	3.17	...	400	
H. C. Reed	50.06	45.27	4.79	600	43.44	6.62	42	152	43.70	6.36	22	152	43.67	6.39	10	152	Candle used, not official.
G. D. McLaughlin	50.50	46.27	4.23	210	45.88	4.62	13	152	46.12	4.38	21	152	46 05	4.45	20	203	4th filtration S. S. 46.15.

Note: Temperature omitted on account of wide variations in results.

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TABLE VI.—STUDY I (c) BERKEFELD CANDLE—QUEBRACHO EXTRACT.

ANALYST.	Total Solids.	Soluble Solids. Official. F. P No. 590.	Ins.	Time. Min.	Soluble Solids. Berkefeld. 1st 100 c.c.	Ins.	Time. Min.	Vac. M.M.	Soluble Solids. Berkefeld. 2nd 100 c.c.	Ins.	Time. Min.	Vac. M.M.	Soluble Solids. Berkefeld. 3rd 100 c.c.	Ins.	Time. Min.	Vac. M M.	
W. H. Teas	90.00	82.77	7.23	...	86.78	3.22	20	40	85.70	4 30	29	40	85.45	4.55	64	40	
E. G. Wilmer	88.10	....	...	...	77.25	10.85	30	40	77.87	10.23	60	40	76.85	11.25	95	40	
J. R. Mardick	90.15	83 78	6 37	27	83.28	6.87	25	40	82.15	8.00	44	40	80.92	9.23	57	40	
F. P. Veitch	90.40	81.76	8.64	120	84.15	6 25	2	40	84.86	5.54	15	40	83.56	6.84	50	40	
J. V. R. Evans	90.25	81.95	8.30	360	86.35	3.90	15	40	86.83	3.42	20	40	86.83	3 42	25	40	
F. H. Small	88.04	80.58	7.46	265	87.59	.45	30	40	....	....	..	...	....	....	..	...	
F. H. Small	88.33	....	...	...	87.06	1.27	15	152	86.74	1.59	25	152	86.06	2.27	30	152	
F W. Alden	87.27	....	...	...	78.10	9.17	25	110	77.84	9 43	25	40	77.36	9.91	40	40	
C. W. Norris	88.14	80.78	7.36	180	87.72	10.42	14	40	87.79	10.35	17	40	87.61	10.53	20	40	
H. T. Wilson	89.95	82.21	7.74	145	81.78	8.17	26	152	81.41	8.54	26	152	82.04	7.91	26	152	4th Filt., S. S., 81.99, candle used. 5th Filt., S. S., 82.56, not official.
A. W. Hoppenstedt	88.79	81.17	7 62	306	86.00	2.79	14	400	84.17	4.62	..	400	82.83	5.96	..	400	
H. C. Reed	87.87	80.03	7.84	240	83.45	4.42	11	152	82.43	5.44	10	152	81.03	6.84	18	152	4th Filt., S. S., 80.83. Candle used, not official.
G. D. McLaughlin.	88.74	81.70	7.04	120	81.71	7.03	14	50	81.91	6.83	7	100	82.34	6.40	12	100	Candle, used, not official.

Note: Temperature omitted on account of wide variations in results.

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TABLE VII.—STUDY I (c) BERKEFELD FILTER CANDLE—HEMLOCK EXTRACT.

ANALYST.	Total Solids.	Soluble Solids. Official. S. S. Per ct.	Ins.	Time. Min.	Soluble Solids. Berkefeld. 1st 100 c.c.	Ins.	Time. Min.	Vac. M.M.	Soluble Solids. Berkefeld. 2nd 100 c.c.	Ins.	Time Min.	Vac. M.M.	Soluble Solids. Berkefeld. 3rd 100 c.c.	Ins.	Time Min.	Vac. M.M.	
W. H. Teas	50.24	46.75	3.49	....	48.94	1.30	15	40	49.18	.96	21	40	48.93	1.29	40	40	
E. G. Wilmer....	50.13	47.59	2.54	160	44.20	5.93	210	40	40.68	9.45	720	40	41 83	8.30	900	40	
J. R. Mardick....	49.20	46.51	2.69	62	45.74	3.46	98	40	45.47	3.73	166	40	45 25	3.96	270	40	
F. P. Veitch	49.99	47.02	2.97	240	41.23	8.76	300	40	43.97	6 02	360	40	....	....	...	...	
J. V. R. Evans...	50.28	47.21	3.07	420	48.38	1.90	15	40	47.99	2.29	20	49	47.92	2.36	25	40	
F. H. Small.....	51 36	47.27	4.09	270	46.70	4.66	330	40	....	...	...	...	....	....	...	...	
F. H. Small.....	51.44	....	...	....	48.06	3.38	85	152	46.05	5 39	285	152	....	....	...	...	
F. W. Alden	50.01	45 14	4.87	1140[1]	....	....	...	...	....	...	...	...	....	....	...	...	[1] Approximate.
F. W. Alden	49 82	....	...	....	35.57	14.25	...	40	....	...	...	...	....	....	...	...	
F. W. Alden	49 93	....	...	....	43.57	6.36	278	60	43.61	6.32	180	50	38.14	11.79	Over night	40	
C. W. Norris.....	50.33	46.38	3.95	240	45.56	4.77	300	40	45.31	5.02	320	40	44.87	5.46	370	40	
M. F. Nichols....	50.64	45.21	5.43	480	48.57	2.07	42	..	46.14	4.50	330	...	46.00	4.64	420	...	Vac. pump left on throughout, running full capacity.
H. T. Wilson	50 48	16.06	4.42	360	45.59	4.89	30	152	45.88	4 60	22	152	45.96	4.52	10	152	4th & 5th Filt. cloudy, candle used. Paper filt. Sli. cloudy, not official.
A. W. Hoppenstedt	50.62	46.41	4.21	600	46.88	3 74	59	400	47.09	3.53	...	400	47.45	3.17	...	400	
H. C. Reed	50.06	45.27	4.79	600	43.44	6.62	42	152	43.70	6.36	22	152	43.67	6.39	10	152	Candle used, not official.
G. D. McLaughlin	50.50	46 27	4.23	210	45.88	4.62	13	152	46.12	4.38	21	152	46 05	4.45	20	203	4th filtration S. S. 46.15.

Note: Temperature omitted on account of wide variations in results.

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The following table showing filtrations of Tannery liquors using the Official Method and the Berkefeld Filter Candle was presented by Mr. McLaughlin:

TABLE VIII.—BERKEFELD FILTER CANDLE—TANNERY LIQUORS.

Using one candle (1⅞″ x 2¼″) for all analyses, brushing between every two or three filtrations and discarding 100 cc. in each case before collecting for evaporation.

	590—S. S.		Berkefeld.		Time.	Vacuum.
Number.	% S. S.	Residue.	% S. S.	Wt. Res.	Minutes.	Inches.
1	3.98	1.2180	3.98	1.2160	26	6
2	3.41	1.0410	5.46	1.0554	30	6
3	6.39	.9844	6.42	.9900	10	6
4	7.19	.7406	7.12	.7340	6	6
5	6.52	.6702	6.61	.6800	6	6
6	7.32	.7554	7.28	.7506	12	6
7	6.51	.6696	6.53	.6714	6	6
8	7.30	.7530	7.30	.7536	15	6
9	7.06	.7274	7.13	.7346	8	6
10	7.52	.7816	7.44	.7676	14	6
11	7.39	.7620	7.43	.7666	10	6
12	9.18	.7152	9.27	.7230	12	6
13	7.65	.7902	7.66	.7910	10	6
14	10.00	.7820	10.05	.7856	10	6
15	7.65	.7898	7.68	.7930	10	6
16	7.54	.7786	7.51	.7760	12	6
Average.	7.04	....	7.05	....	12.3	..

Note: The 590 S. S. filtrations average 30 minutes.

Mr. McLaughlin further presents results included in the following tables:

TABLE IX.—SPENT OAK BARK.

	Wt. Res.	S. S. %	Time. Min.	Vacuum.	Appearance.
1st 100 cc.....	.5830	4.93	18	2″	Brilliant
2nd "	.5780	4.89	15	2″	"
3rd "	.5795	4.90	17	2″	"
590 S. S.....	.5697	4.81	40	..	"

TABLE X.—CHESTNUT EXTRACT.

	Wt. Res	S. S. %	Time. Min.	Vacuum.	Appearance.
Total Solids	.5870	36.62	..	...	.....
1st 100 cc.....	.5784	36.08	35	4″	Brilliant
2nd "	.5783	36.08	33	4″	"
3rd "	.5814	36.27	12	8″	"
4th "	.5808	36.23	12	8″	"
5th "	.5828	36.36	6	12″	"
6th "	.5823	36.33	11	12″	"
7th "	.5817	36.31	5	16″	"
590 S. S.....	.5776	36.00	40	...	"

Mr. McLaughlin says, "The figures in last table are an attempt to show the difference, if any, on filtrations with high and low vacuum."

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COLLABORATORS' NOTES RELATIVE TO STUDY I (c).

E. G. Wilmer.

"I should like to note that owing to the slow filtration of the hemlock extract, it is almost impossible to prevent evaporation, which would materially effect the results."

J. R. Mardick.

"Berkefeld candle works pretty well with light tannin material like Quebracho, provided that the pressure is kept constant. Forty mm. pressure would not do, it will require 80 mm. vacuum constant. By *constant,* I mean not to shut off the pressure by cock as directed. This filtration method will not work well with heavy extracts like Hemlock. As you will notice, there is a gradual decrease in each successive soluble solids filtration.

Again, the candle is pretty nearly covered with solid matter after the first 150 cc. filtration, and in the second and third 100 cc., it requires nearly complete vacuum in the case of Hemlock.

By Berkefeld method filtration the solutions are much clearer with each successive filtration, but the per cent soluble matter decreases with the clearness.

For a technical laboratory, where a large amount of work is done, this method is very slow, and impracticable as well as unhandy. Again, the extract manufacturers would not like this method for obvious reasons (less soluble solids), as from the standpoint of tanners such method of filtration (under pressure) is entirely out of discussion."

F. P. Veitch.

"The temperature of filtration was from 25 to 28°. The results reported for 2nd 100 cc., on quebracho with the candle, are, I fear, erroneous, but I had neither the time nor material to repeat.

The required 150 cc. were first rejected in all cases.

Three or four years ago I tried this scheme, using a closer pored candle than the Berkefeld, but the results were so low that I abandoned further trials. The quebracho solutions, which had passed the candle, were not bright, but decidedly opaque."

F. H. Small.

"The filter candle will not separate out the reds from the quebracho until a very large quantity of liquor has been filtered and the candle become clogged. On the other hand the reds of the

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hemlock clog the filter rapidly and then filtration proceeds very slowly even when a brush is used occasionally on the candle. I found further that care was very necessary in using a brush, as otherwise the candle again allowed reds to pass through.

For that matter, I did not get very satisfactory results with the official method, as it did not give perfectly clear filtrate in several instances. This is in line with all my past experience and is why I have so persistently advocated a time contact method or some similar process. On these two extracts my assistant and myself by my laboratory method got results for insolubles as follows, quebracho 7.42, 7.52, 7.55. Hemlock 4.98, 5.35, 5.22, while with equal care the official method gave quebracho 7.35, 6.98, 7.46, Hemlock 4.61, 4.43, 4.09, and several of these latter filtrates were not clear.

These two extracts seem unusually well adapted to showing up the deficiencies of the Berkefeld filter candle, and I am more than ever convinced that we have nothing to gain by adopting it."

F. W. Alden.

"I regret that I am unable to make a satisfactory report on the use of the Berkefeld filter candle.

The hemlock extract filtered very slowly indeed, and although the candle was brushed and the vacuum repeatedly renewed, the only way seemed to be to let it take its time. The worst of it was that the solutions had to stand much longer than they should have before they were filtered. I undertook to do duplicates with the hemlock which delayed the work still more. The filtration was so slow that I did not attempt to control the temperature.

Hemlock Extract.

The filtration was very rapid at first, and the filtrate very cloudy so that much more than 150 cc. passed through before a clear filtrate was obtained.

Several hours were required to obtain 150 cc. of clear liquor.

It seems that the candle does absorb considerable, especially when in contact with the liquor for a long time.

In the results with quebracho, the extract solution had stood four days. The filtrate did not become clear until 750 cc. had passed through, all of which was returned excepting the first 150 cc."

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C. W. Norris.

"I found that by using only a 500 cc. flask, I could get only 30 to 40 cc. of filtrate before the pressure would diminish from 40 mm. to 0. So instead of renewing the pressure a number of times, I inserted a *six* liter flask; and the pressure would diminish from 40 to 30 mm., during each 100 cc., and thus keeping a more steady pressure throughout.

With the quebracho, filtrates were as cloudy as before filtration.

With the hemlock, filtrates were perfectly clear. The candle was brushed lightly with toothbrush three times during each filtration. Because of slow filtration only 1st 100 cc. could be obtained first day. The 2nd and 3rd 100 cc. were filtered the following day. Temperature was constant throughout."

M. F. Nicholas.

"I had no vacuum gauge, but merely attached filter pump and ran it to full capacity. My filter had a flaw and got to leaking on making my test on quebracho and would not filter at all, so cannot give you any report on this sample.

With my experience with Berkefeld filter I cannot say that I am much in favor of it."

F. R. Mosbaugh.

"Found it impossible to obtain clear filtrate with Berkefeld filter, the only approach to clearness being when filter was clogged, and upon slightly brushing candle, the cloudiness would reappear. Think it must have been in some way defective."

H. T. Wilson.

"Could not make the small candle work to advantage. Clogs up too quickly and is too slow to be practical on hemlock and quebracho. In all cases I ran the vacuum up to 6" and did not touch cocks until filtration was complete. Hence there could be no evaporation. Do not favor vacuum as low as 40 mm., because it is scarcely enough for a complete filtration and is very slow. I have three other determinations on another Quebracho solution, which run .4966, .4947 and .4943 = 82.46%, 82.15% and 82.09% against .4930 = 81.87% for No. 590 paper."

A. W. Hoppenstedt.

"All the filtrates from the paper were clear, while those from the candle filter were cloudy.

In the Quebracho extract the filtrates from the candle filter be-

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came gradually clearer as the filter became more clogged up with reds and filtered slower. I think this accounts for the results here.

In the Hemlock extract the first 100 cc. went through so slowly that I began to use a brush on the second 100 cc., and at once I could notice that the filtrate become more cloudy. On the Quebracho extract I did not use a brush at all. This I think accounts for these results showing just the opposite from the Quebracho extract.

In my opinion the boiling of the filter candle with hydrochloric acid in order to remove the iron, etc., rendered the same too porous thereby destroying its filtering power. The absorption of tannin by the candle filter, I think, is extremely little.

From the above work and from the work I did previously I feel convinced that the candle filter method as it now stands is of little value."

Chas. Eachus.

"I tried the candle filter on the two extracts and found that it did not filter clear, until it became so clogged up with reds as to render it worthless, and as soon as the filter was scrubbed with a toothbrush the filtrate became cloudy again. My results are worthless, I do not wish to report them, and I do not intend to use the candle filter, unless the preponderance of opinion of the chemists as the annual meeting is in favor of it."

G. D. McLaughlin.

"Candle supplied by Referee too small for Hemlock solution, too smal surface exposed which made time to filter too long."

The results as shown by the tables and the remarks of the collaborators are distinctly unfavorable to the Berkefeld filter candle filtration. Mr. Wilson, who in his report as Chairman, gives some excellent results with the filter candle, confesses that he was unable to use to any advantage, the candle submitted by the Referee.

The general concensus of opinion is that only by the stoppage of the pores of the candle with the insolubles of the solution, can clear filtrates be obtained, and when thus obtained, filtration is too slow to be practicable, and when hastened by brushing, cloudy filtrates result.

The Referee has made certain tests which seem to indicate a gradual absorption of tannin matters by the filter candle. These results are included in the table given below.

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TABLE XI.—TABLE SHOWING SOLUBLE SOLIDS LOSS WITH BERKEFELD FILTER CANDLE.

	Paper.	Berkefeld.																	
	Official.	1st 100 c.c.	Time Min.	2nd 100 c.c.	Time Min.	3rd 100 c.c.	Time Min.	4th 100 c c.	Time Min.	5th 100 c.c.	Time Min.	6th 100 c c.	Time Min.	7th 100 c.c.	Time Min.	8th 100 c.c.	Time Min.	9th 100 c.c.	Time Min.
Chestnut Extract (1)	40.38	40.35	4	40.53	4	40.54	4	40.45	4	40.11	15	39.11	25	....	..	....	..	....	..
Hemlock Extract...	40.76	37.39	60	39.82	30	....	..	....	..	....	..	...	..	....	..	....	..	....	..
Quebracho Extract · Unclarified.	74.70	76.70	3	76.10	3	75.31	10	....	..	....	..	....	..	....	..	....	..	....	..
Quebracho Extract · Clarified (1)	....	49.02	5	49.03	7	48.59	9	48.28	12	48.04	15	47.67	20	47.33	22	47.11	25	47.70[1]	10
" (2) ...	47.63	48.11	..	47.35	..	47.01	..	....	..	....	..	....	.	....	..	....	..	....	..
" (3) ...	47.92	48.23	..	47.49	..	47.41	..	....	..	....	..	....	..	....	..	....	..	....	..
" (4) ...	46.69	47.13	..	46 52	..	46.36	..	....	..	....	..	....	..	....	..	....	..	....	..
" (5) ...	45.70	45.63	..	45.47	..	47.12	10	....	..	....	..	....	..	....	..	....	..	....	..
" (6) ...	45.93	47.20	..	46.05	..	45.70	..	....	..	....	..	....	..	....	..	...	..	....	..
" (7) ...	48.00	48.72	..	47.33	..	46.54	..	46.70	..	46.25	..	46.22	..	46.18	..	46.05	..	....	..
Chestnut Extract (2)	....	39.52	5	39.38	5	39.95	5	39.73	5	39.87	5	39.88	5	38.44[2]	5	39.91	5	....	..

[1] Brushed before filtering. [2] Candle stood in contact with solution 1½ hours before filtering.

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You will observe that almost invariably the first 100 cc. passing through the filter candle gives higher soluble solids than shown by our present official method of paper and kaolin filtration.

The report of the I. A. L. T. C. commission, upon which was based the adoption of the Berkefeld filter candle, apparently shows that no further attempts were made in their investigation than the collection of this first portion for soluble solids determination, after rejecting the prescribed amount of filtrate. It is true that work had been done upon clear tannin solutions which would indicate no absorption of tannin by the candle filter, and it may be that the candle itself is not directly responsible for loss of tannin through its own absorption but that the coating of insoluble has some influence not explained. At any rate the constant decrease in soluble solids is plainly apparent, and the element of time has an important bearing.

The results obtained by Mr. McLaughlin on tannery liquors, using the filter candle, are very good, but it must not be lost sight of that the insolubles of liquors are of entirely different character from those of extracts, and do not clog the pores of either the paper or candle. Moreover, the average soluble solids by candle and paper filtration would indicate as much absorption of tannin by one as the other, as there is but a difference of .01% in favor of the candle. In Table 10, Chestnut extract, Mr. McLaughlin's results in a measure bear out the contention of the Referee relative to the influence of time.

It is the experience of the Referee that the candle filter shows as much, if not greater, absorption than the filter paper when the time for filtration is equal in both cases, or, conversely, it is only owing to rapidity of filter candle filtration that higher soluble solids results are shown by the method than by paper filtration.

Mr. Hoppenstedt suggests that the treatment with hydrochloric acid for the removal of iron may have softened the candle and made it more porous. The Referee, in order to determine the value of this suggestion, used a candle which had been washed with hot water only, but met with all the difficulties experienced with the acid treated candle.

There can be no question from the collaborative results on the Berkefeld filter candle, as to its adoption by this Association.

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NON-TANNINS AND THE HIDE POWDER QUESTION.

As shown by the tables, the non-tannin results are, on the whole, very excellent indeed.

The Referee considers the reports of the Committee on the Chroming of Hide Powder, on the questions of the moisture content of the wet chromed hide and of adding all the chrome alum at once instead of in two portions, to be sufficiently conclusive, and recommends that these changes be made in the present official method.

Mr. Small has offered a solution of the question as to the reason of lower and more uniform non-tannin results when the water content of the wet hide falls between 70 and 75%. He has found that, if, after shaking, the hide powder is freed from solution by light squeezing and then placed in a press and subjected to severe squeezing, the two portions of liquor obtained when filtered, measured, dried and weighed, give different non-tannin figures, the latter being appreciably lower than the former. It would appear that the solution more closely retained by the hide powder, was more fully depleted of absorbable matters. When the moisture content of the hide powder approximates that to which it can be squeezed by the hand, lower non-tannins result than when the hide powder, previous to use, is squeezed to a low moisture content which cannot be attained by hand squeezing after shaking with the tannin solution.

It would not seen advisable, judging from the report of the Hide Powder Committe, to alter the present limits of from 12-13 grams of dry hide in the wet hide used for detannizing 200 cc., of the tannin solution as per present official method, nor to reduce the time for shaking, nor to chrome for a shorter period.

The dry chromed hide powder failed to give the result that was expected of it. There seems to have been a gradual deterioration in its power of absorbing tannin. Very possibly due to the reason suggested by Mr. Alsop, to wit: loss of absorption power by drying and hardening.

The Referee received from Mr. Alden, of the Pfister and Vogel Leather Company, a sample of chromed hide powder, practically chrome leather, which he tested against the wet chromed powder. The comparison is given in a table below.

It must be understood, however, that this powder requires

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washing, and must therefore be used in the wet state, with a moisture correction, in a manner exactly similar to our present mode of operation with, of course, the omission of the chroming.

It is claimed for a powder of this nature, that better duplication will be obtained in Non-tannin item than when hide powder is but partially chromed as with our present method.

It is a subject that would well repay investigation and this fully chromed powder seems to have lost in chroming little, if any, of its absorbability for tannin.

TABLE XII.—TABLE SHOWING ALDEN'S CHROMED HIDE POWDER VS. WET CHROMED HIDE POWDER.

			N. T.	Wet Hide.	H P. Moisture.	Dry Hide.
Quebracho Extract.....	(1)	Wet Chromed	11.08	48	73	13.0
		Alden's	12.25	44.5	75	11.13
	(2)	Wet Chromed	10.56	46	72.5	12.65
		Alden's	11.03	47	73.6	12.41
Hemlock Extract	(1)	Wet Chromed	20.41	48	73	13.0
		Alden's	20.92	44.5	75	11.13
	(2)	Wet Chromed	19.19	46	72.5	12.65
		Alden's	18.86	47	73.6	12.41
Chestnut Extract	(1)	Wet Chromed	11.01	48	73	13.0
		Alden's	11.75	44.5	75	11.13
	(2)	Wet Chromed	11.26	46	72.5	12.65
		Alden's	11.27	47	73.6	12.41
Oak Extract.....	(1)	Wet Chromed	20.52	48	73	13.0
		Alden's	20.65	44.5	75	11.13
	(2)	Wet Chromed	20 46	46	72.5	12.65
		Alden's	19.87	47	73.6	12.41
Spent Oak Liquor.....		Wet Chromed	.42	6	73	1.62
		Alden's	.43	5 5	75	1.38
Spent Hemlock Liquor...		Wet Chromed	.88	11.5	73	3.1
		Alden's	.91	10.0	75	2.5

EVAPORATION AND DRYING.

That some basis might be had from which to draw conclusions as to the relative value of various systems of evaporation and drying, the Referee has compiled a table from the extract analyses tables showing the total solids comparisons by the three principal methods employed.

The least variation in the case of the Quebracho extract was with the steam oven, and with the Hemlock extract the combined evaporator and dryer showed to best advantage. It must be considered, however, that the chance of variation from the

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TABLE XIII.—TABLE SHOWING COMPARATIVE RESULTS FROM METHODS OF EVAPORATION AND DRYING.

Method and Extract.	"A"—Quebracho.			"B"—Quebracho.			"A"—Hemlock.			"B"—Hemlock.		
Method of Evaporation and Drying	E. & D.	W.O.	S.O.	E. & D.	W. O.	S. O.	E. & D.	W. O.	S. O.	E. &. D.	W. O.	S. O.
Average Total Solids	89.25	90.19	88.73	89.27	90.02	88.88	49.88	50 14	50.04	49.88	50.01	50.04
Maximum	89.91	91.98	89.22	89.95	90.62	89.07	50.09	49.81	50.56	50.20	50.47	50.56
Minimum	88.66	88.74	88.33	88.75	88.74	88.50	49.50	49.70	49.71	49.67	49.70	49.71
Greatest Difference	1.25	3.24	.89	1.20	1.88	.57	.59	1.11	.85	.53	.77	.85
Number Analysts represented	8	6	4	8	6	4	8	6	4	8	6	4

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evaporator and dryer was greater owing to the larger number of analyses represented by this method. The water oven apparently gives more variable results, owing, no doubt, to the greater variation in the temperature. As a matter of fact, the combined evaporator and dryer is, in a sense, not widely different from the steam oven, and the former can very readily be converted into the latter. Assuming that both are of equal merit in results given, the advantage of ease of manipulation lies with the combined evaporator and dryer. The apparatus has been used in the laboratory of the Referee for nearly a year, and he can vouch for its efficiency and reliability. It is believed that other analysts who have installed the apparatus will bear out this statement.

The Referee is so convinced of the merits of this method of evaporation and drying tannin residues, that he has decided to recommend the provisional adoption of the combined evaporator and dryer.

It is possible that the discrepancies in total solids figures may be in part accountable to incorrectly standardized pipettes. It may seem superfluous to direct attention to a contingency of this kind, but the Referee advises that pipettes be carefully re-graduated when received.

Before leaving Study I, the Referee wishes to ask a single question in respect to Mr. Mardick's so-called "Tanner's Method" of tannin analysis and his more recent method in which acid is added to the extract solution. Mr. Mardick has claimed for his tanner's method that it gives results in all respects comparable with those obtained in tannery practice. If such is the case, with what are his results from his acid-addition method, where he shows considerably less tannin than by his tanner's method, comparable?

STUDY II.

ANALYSIS OF LIQUORS.

The Referee suggested to Mr. Small, Chairman of the "Liquor" Committee, that his committee should work with the view of producing a suitable provisional method of analysis, and to study the effect of the acids of liquors upon the analysis.

In answer to the request, the chairman submitted the following report:

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COMMITTEE REPORT ON ANALYSIS OF SPENT LIQUORS.

The composition of spent liquors is peculiar in that the ratio of the tannin to the matters not tannin,—glucosides, acids, inorganic matter, etc., is very small. This raises the question whether the official method of analysis is applicable to such liquors,and if not how the method can be modified to meet the peculiarities of the case. Certain preliminary experiments were made by the chairman to study this question, with the results as follows:

Two spent oak liquors were analyzed at three different dilutions, the liquor being diluted with water at 80° C., and allowed to cool slowly, also two stronger liquors were analyzed at two different dilutions made as above.

Barkometer	18.2			18.4		
	1 Liq. Wat.	2 Liq. Wat.	3 Liq. Wat.	1 Liq. Wat.	2 Liq. Wat.	3 Liq. Wat.
Dilution	1000—0	600—400	190—810	1000—0	600—400	190—810
Total Solids ..	4.31	4.30	4.34	4.42	4.39	4.43
Soluble Solids	4.24	4.19	4.23	4.32	4.30	4.33
Non-tannin...	4.14	3.88	3.68	4.24	4.01	3.78
Tannin	.10	.31	.55	.08	.29	.55
Insolubles	.07	.11	.11	.10	.09	.10

Hide Powder per 100 cc., 18.8 gms., containing 70.5% moisture.

Barkometer.	33.4		28.8	
	1 Liq. Water.	2 Liq. Water.	1 Liq. Water.	2 Liq. Water.
Dilution	220—780	100—900	250—750	120—880
Total Solids	8 17	8.16	7.07	7.07
Soluble Solids	8.08	8.01	7.01	6.96
Non-tannin	6.09	5.84	5.78	5.58
Tannin.....	1.99	2.17	1.23	1.38
Insolubles.....	.09	.15	.06	.11

Hide Powder per 100 cc., 18.8 gms., containing 70.5% moisture.

The total solids residues from the evaporation of 100 cc. of spent liquor in the first table at dilutions 1 and 2 were respectively 4.39 grams, 4.50 grams, and 2.62 grams, and 2.68 grams. These are too large for satisfactory drying by the official method. Further, and as was to be expected, varying the dilution but using the same amount of hide powder, resulted in varying the non-tannin percentage greatly. Dilution 1 in each case corresponded most closely to the official and consequently represented most nearly theofficial ratio of hide powder to tannin. At this dilution filtrations were exceedingly slow, frequently requiring many hours. At the higher dilutions, the ratio of hide powder to tannin was unquestionably excessive. There was further some question as to whether the process of dilution did not put in solu-

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tion matter insoluble in the liquor at the original concentration. A slight tendency in this direction is shown by the various analyses in this report, but it is so small as to be practically negligible. The results obtained by Messrs. Cushing and Teas are similar, though from previous experience, both feel that there is some solution of insolubles by the dilution.

In order to find whether a dilute solution and less hide powder or one made up of official strength was the more desirable, the following analyses were made:

Barkometer.	1	2 18.7°.	3	1	2 18.7°.	3	4
Dilution	1000 cc. Liq.—0 Wat.			200 cc. Liq.—800 cc. Water.			
Total Solids	4.465	....	....	4.448	....	....	....
Soluble Solids	4.341	....	...	4.348	....	....	....
Insolubles	.124	....	....	.100	....	....	....
Non-tannin	4.114	4.113	4.157	3.729	3.756	3.856	4.020
Tannin	.227	.228	.184	.619	.592	.492	.328
Hide Powder to 100 cc	22.7g.	16.4g.	10.cg.	22.7g.	16 4g.	10.0g.	3.7g.
Moisture in H. P.	....	....	72.3%	....	....	....	...
Abs. dry H. P.	6.28g.	4.54g.	2.76g.	6.28g.	4.54g.	2.76g.	1.02g.

Barkometer.	1	2 18.9°.	3	1	2 18 9°.	3	4
Dilution	1000 cc. Liq.—0 Wat.			200 cc. Liq.—800 cc. Water.			
Total Solids	4.553	....	....	4.549	....	....	....
Soluble Solids	4.442	....	....	4..461	....	....	....
Insolubles	.111	....	....	.088	....	....	....
Non-tannin	4.253	4.254	4.299	3.856	3.910	3.995	4.166
Tannin	.189	.188	.143	.605	.551	.466	.295
Hide Powder to 100 cc.	22.7g.	16.4g.	10.0g.	22.7g.	16.4g.	10.0g.	3.7g.
Moisture in H. P.			72.3%.				

No. 4 of the dilute solution in which approximately the official ratio of hide powder to tannin is kept, shows a larger absorption than the corresponding No. 1 at full strength. That the tannin figure in the latter case is amply large is seen from the fact that all the non-tannin filtrates even when only 10 grams of wet hide were used, gave absolutely no test for tannin with a gelatine-salt solution.

High dilutions require small amounts of hide powder, in which case variations of a fraction of a gram in the amount used cause relatively large variations in the amount of matter absorbed, while at full strength little change is produced by a change of several grams in the amount of wet hide powder. For these reasons it seems desirable to use for analysis dilutions containing as near as practicable the official amount of tannin, namely .35-.45 grams per 100 cc. That more matter will be absorbed by this

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procedure than is precipitable by a gelatine-salt solution is undoubted.

To form some idea of the excess, a liquor at approximately official strength was treated with lessening amounts of hide powder in order to find how small an amount could be used and yet yield a non-tannin filtrate free from matter precipitable by gelatine-salt solution.

Barkometer.	1	2	18.4°. 3	4	1	2	17.1°. 3	4
Dilution	1000 cc. Liq.—0 Water.				1000 cc. Liq.—0 Water.			
Total Solids	4.392	....	....	....	4.021	....	....	....
Soluble Solids	4.312	....	....	....	3.955	....	....	....
Insolubles	.080	....	....	....	.066	....	....	....
Non-tannin	4.063	4.092	4.127	4.210[1]	3.770	3.805	3.814	3.867[1]
Tannin	.249	.220	.185	.102	.185	.150	.141	.088
Hide Powder	22.2	15.9	9.5	3.2	22.2	15.9	9.5	3.2
Moisture in H. P.			72.1%.					

[1] These gave test for tannin with gelatine-salt solution.

Clearly when a spent liquor is analyzed according to the official method, the hide powder absorbs a considerable amount of the matter not tannin. That this is true in general even when fresh extract solutions are analyzed is doubtless recognized by all; but even so, all the matter not tannin in the liquor and absorbable by hide powder is not then removed. To show this 300 cc. of a 20 Bk. liquor were shaken 10 minutes with 60 grams of wet hide powder containing 70.3% moisture. The hide powder was filtered out, 20 cc. of clear filtrate were evaporated and 200 cc. of the remaining were shaken with 42 grams of wet hide powder. The hide powder was again filtered out, 20 cc. of clear filtrate evaporated and 100 cc. of the remaining were shaken with 21 grams of wet hide powder. Twenty cc. of the clear filtrate from this were also evaporated. The results obtained were as follows:

	1	2
Soluble solids in original solution	4.702%	4.548%
First non-tannin residue	4.438	4.301
Matter absorbed by hide powder from original solution	.264	.247
First non-tannin residue	4.428%	4.301%
Second " "	4.322	4.180
Matter absorbed by hide powder from first non-tannin residue	.166	.121
Second non-tannin residue	4.322%	4.180%
Third " "	4.300	4.134
Matter absorbed by hide powder from second non-tannin residue	.022	.046

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In other words, fresh hide will still absorb considerable matter from a solution after the solution has yielded up much more than the tannin content as shown by gelatine-salt test.

In continuation of the above test, the tanned hide powder from the first shaking was carefully weighed, then put in a glass and shaken with 200 cc. of water. The tanned hide from the second shaking was similarly treated with 100 cc. of water. In each case 50 cc. of clear filtrate was evaporated. It was hoped to discover whether there was a combination between the matter absorbed and the hide or whether the matter was so loosely held that it could be readily washed out. In each case, however, the water after the shaking contained only so much matter as could be accounted for by the liquor present in the tanned hide powder when put in the water.

No great reliance is to be placed on these results since they rest wholly on the assumption that after hide powder has been shaken with tan liquor the composition of the liquor surrounding the hide powder and that present in the hide powder, but which cannot be removed by pressing, is identical. The chairman feels grave doubts of the truth of this assumption.

Until there is an agreement as to what tannin analysis means, whether it is an attempt at a determination of tannic acid, of matter absorbable by hide, of matter not precipitable by gelatine-salt solution, or what not,—the chairman feels that the closer we stick to a uniform method of analysis the more comparable our results are likely to be.

Certain further experiments were undertaken with a view to ascertaining the effect of the time of shaking of hide powder and liquor,—originally in hopes that the hide powder would remove tannin first and so a short shaking would suffice; later to discover whether a prolonged shaking with a smaller amount of hide powder might yield the same absorption as a shorter shaking with more. The results obtained were as follows:

Barkometer	18.2	18 4	28 8	33.4	54.1	56.1
Non-tannin, 3 minutes shaking	3.92	4.04	5.85	5 22	6.86	6.82
Non-tannin, 10 " "	3.88	4.01	5.78	6.09	6.58	6.53

Barkometer	18.4		17.1	
Non-tannin, 10 min. shaking	4.127	4.210	3.814	3.867
Non-tannin, 30 " "	4.126	4.202	3.821	3.871
Hide powder absolutely dry	4.43 gms.	2.65 gms.	4.43 gms.	2.65 gms.

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Three minutes does not produce a maximum absorption and as the absorption then becomes a function of the time, it is essential in order to secure concordant results that the time be regulated to a fraction of a minute, a proposition which is hardly feasible. In 10 minutes a maximum absorption has been reached, hence the futility of hoping by prolonged shaking and less hide powder to reach the same result as with more hide powder. In general the amount of matter absorbed is a direct function of the amount of hide powder employed and after absorption has reached a maximum, which occurs in 10 minutes or less, it is independent of the time.

The chairman would here acknowledge his indebtedness to his assistant, Miss Worthen, for valuable aid in the experimental part of the work.

This preliminary work led the chairman to believe that spent liquors should be analyzed at the same dilution (.35-.45 grams tannin per 100 cc.) as extract solutions,—that a smaller aliquot part than 100 cc. should be evaporated and that the soluble solids determination should be modified. Samples of seven representative spent liquors were consequently sent to the members of the committee with instructions as follows:

"Make up solutions to contain from .35 to .45 grams tannin per 100 cc., using water at 80° C. for the dilution. (Liquor No. 1 is too dilute to do this so proceed with it at full strength and in determining non-tannins use only one-half of the required amount of hide powder). If 100 cc. of these solutions be evaporated the residues will be too large for satisfactory drying by the official method, hence in each case evaporate an aliquot part that will give a residue for soluble solids of from .7 to .8 grams approximately. Evaporate the residue in 3" dishes and dry as usual. Determine the soluble solids as usual except that after rejecting 150 cc. you will collect only so many cubic centimeters as needed. It may be preferable to leave the liquor in contact with kaolin and filter paper a definite time instead of rejecting a definite number of cubic centimeters. Study of this point will be welcomed. Determine non-tannins by shaking 10 minutes with an amount of wet chromed hide powder equivalent to from 12 to 13 grams absolutely dry. Test the non-tannin filtrates in each case for tannin with a gelatine-salt solution.

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Appended are the dilution and number of cubic centimeters to be evaporated for each liquor, which the chairman thinks will satisfy the requirements of the above method. It is perhaps needless to suggest that special attention be given to the accuracy of the pipettes used since a slight error in the pipetting is largely magnified when using solutions of such comparatively high concentration.

	No. of Sample.	Dilution. Liquor.	Water.	CC. to be Evaporated.
1	Spent Union Liquor	1000	0	50
2	Nearly Spent Union Liquor	800	200	40
3	Spent Acid Hemlock	600	400	30
4	Nearly Spent Acid Hemlock	400	600	35
5	Spent Oak	1000	0	20
6	Nearly Spent Oak	700	300	20
7	Oak Liquor	225	775	40

The results obtained by Messrs. Teas, Cushing and Small are tabulated herewith, as also results obtained by Mr. Teas by his laboratory method, an account of which will be found later in the report.

	Teas.	No. 1. Cushing.	Small.	Teas.	No. 2. Cushing.	Small.
Total Solids	1.675	1.63	1.651	2.580	2.52	2.524
Soluble Solids..	1.608	1.55	1.574	2.381	2.32	2.334
Non-tannins....	1.469	1.42	1.447	1.703	1.67	1.670
Tannin.....	.139	.13	.127	.678	.65	.664
Insolubles.....	.067	.08	.077	.199	.20	.190
		No. 3.			No. 4.	
Total Solids	3.772	3.70	3.694	5.809	5.54	5.543
Soluble Solids..	3.736	3.61	3.629	5.700	5.42	5.481
Non-tannins....	2.946	2.85	2.862	4.732[1]	4.60	4.629
Tannin.....	.790	.76	.767	.968	.82	.852
Insolubles.....	.036	.09	.065	.109	.12	.062

	Teas.	No. 5. Cushing.	Small.	Teas.	No. 6. Cushing.	Small.	Teas.	No. 7. Cushing.	Small.
Total Solids	4.21	4.10	4.133	5.603	5.37	5.404	8.700	8.30	8.306
Soluble Solids..	4.08	3.97	4.011	5.446	5.28	5.288	8.578	8.13	8.169
Non-tannins	3.82[1]	3.68	3.732	4.673[1]	4.59	4.578	6.327	6.08	6.048
Tannin.....	.26	.29	.279	.773	.69	.710	2.251	2.05	2.121
Insolubles.....	.13	.13	.122	.157	.09	.116	.122	.17	.137

[1] Gave test for tannin.

Results by Mr. Teas Laboratory Method.

	1	2	3	4	5	6	7
Total Solids....	1.710	2.620	3.861	5.833	4.33	5.69	8.868
Soluble Solids..	1.596	2.396	3.758	5.733	4.06	5.48	8.672
Non-tannin	1.445	1.715	2.947	4.628	3.51	4.45	6.362
Tannin	.151	.681	.811	1.105	.55	1.03	2.310
Insolubles	.114	.224	.103	.100	.27	.21	.196

Mr. Mardick submitted the following report, but as the chairman has been unable to prove the figuring by any mathematics

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known to him, and as the values at best are widely different from those obtained by the rest of the committee, it was thought folly to include them in the above summary.

	1	2	3	4	5	6	7
Total Solids..	"Did not make any total solids determination, and could not see any reason for it."						
Soluble Solids	.7362	.7041	.6361	.7392	.7224	.6720	.6907
Non-tannins[2].	.5762	.3939	.4051	.5109	.5520	.4744	.4505
Tannin, %...	.24	.82	.89	.98	.50	.98	2.00

[2] Without hide powder correction.

Mr. Mardick recommends in place of the above method, his maceration method, and sends his results by this method.

	1	2	3	4	5	6	7
Soluble Solids	.7360	.5656	.6110	.6944	.6553	.4325	.5055
Non-tannin[1].	.5758	.3426	.4209	.5633	.5353	.3529	.2427
Tannin, %...	.24	.89	.96	1.05	.72	1.38	1.94
Diluted.....	0	2	3	4	3	4	4

Ten grams of moist hide powder used and 50 cc. evaporated in each case. Time of maceration, 1 hour. Four times stirred.

[1] Corrected.

With regard to the method outlined by the chairman, Mr. Mardick, says: "I regard the method of dilution and quantity of evaporation simply tortuous and unpractical. Sour liquors cannot be treated like sweet extract liquors because the acid holds back the tannin, hence the necessity of dilution to a certain degree, say 6° Bk. Strong sour liquors dissolve a part of the unreduced chrome in the hide powder which results as large non-tannin residue. Strong sour liquors have solvent action on hide powder. Qualitative tests made on the tannin residue confirm these statements."

Some of these objections of Mr. Mardick are answered in the course of this report. As to the presence of Chromium in the non-tannin residue, the chairman made careful ash determinations of the non-tannin residues from two strong sour spent liquors, and in neither case was he able to detect the presence of chromium by any of the ordinary qualitative tests. Mr. Mardick's results might well have been due to imperfect washing of his hide powder.

Mr. Cushing says, with regard to the proposed method, "It seems to me to be thoroughly practical with possibly one exception,—whether dilution with water at 80° C. might not carry into solution material insoluble in the original liquor. ———— I made a rather incomplete comparison between the method of hot

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dilution and a method by which the total solids and soluble solids were determined from the original liquor. These tests did not bear out this contention, notably in the case of No. 7, which would be more inclined to show any solutions of reds on account of its high dilution."

His report on this subject reads as follows: "The following table gives some results upon these liquors by varying the procedure to some extent. The total and soluble solids were in this case determined from the undiluted liquor, the dilution for non-tannin determination being made with water at 20° C. The proper amount of liquor was measured into the shaking jar, diluting with water to a total volume of 200 cc. The object of making the comparison was to determine if possible if any substances insoluble in the original liquor were carried into solution by diluting with water at 80° C., as recommended in the first tests. Owing to the limited time at his disposal, the writer did not go into this question as fully as he should have done, being obliged to use for analysis the original samples which had already been tested, on account of the data already accumulated, although in most cases there was not sufficient quantity of liquor to make complete analyses.

No.	Total Solids.	Soluble Solids.	Insoluables.	Non-tannins.	Tannins.	*	Diluted for N. T.	CC. Evap. N. T.
2	....	....	....	1.63	.. .	....	125 L— 75 W	40
3	3.72	....	....	2.84	....	20	100 L—100 W	30
4	5.55	5.43	.12	4.66	.77	10	100 L—100 W	35
6	5.40	5.25	.15	....	....	10	....	....
7	8.29	8.12	.17	6.10	2.02	5	40 L— 60 W	40

* cc evaporated in determining total solids and soluble solids.

The results do not appear to indicate that any serious variation was caused by solution of reds in the case of the hot dilution. It would be interesting to determine this point more definitely, using a fresh liquor of high concentration, containing a considerable proportion of reds.

It was suggested by the chairman that the following method for determining soluble solids be tested:—Add one gram of kaolin to 75 cc. of liquor, pour on single pleated filter, collect filtrate and return through filter for one hour, keeping funnel well filled until toward the end of the hour. Pour out any liquor left in the funnel, let drain a few moments; reject the first 10 to 15 cc. of filtrate or until it is clear, then collect for evaporation,

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keeping funnel full. The above procedure was followed out with samples No. 6 and No. 7. Duplicates of No. 6 gave in each case 5.25%; triplicates of No. 7 gave 8.10, 8.07, 8.09. These results were obtained from the undiluted liquor. The chairman states that he finds this method of time-contact with the filter more satisfactory than the official method. The writer is of the same opinion, having followed a method for control work which is quite similar. This consists in passing 75 cc. of liquor through the filter, keeping funnel as full as possible, then drawing out the liquor in the filter with a pipette, removing practically all in this way, filling with fresh liquor, rejecting the first filtrate if cloudy and collecting the necessary amount.

Mr. Teas says: "I do not think the method you suggest has any advantages over our method as regards results obtained. As regards time and trouble, our method has decided advantages."

The method to which Mr. Teas refers is as follows:

Total Solids....	Shake well; remove 10 cc., evaporate and dry in dish 2¼" D.		
Soluble Solids..	To 200 cc. add kaolin, stir, filter through double Prat-Dumas paper, rejecting filtrate of first half hour, then collecting 10 cc. and drying as above.		
			L. W.
Non-tannin	Dilute liquors thus:	20 Bk. and higher	50—500
		10 Bk.—20 Bk.	100—500
		Below 10 Bk.	200—500
	Use amounts of hide powder from 20 to 45 grams per 200 cc., depending upon the strength and nature of the liquor. Shake 3 minutes, add kaolin, stir, filter through double Prat-Dumas paper. Collect 100 cc. for evaporation.		

Mr. Teas continues: "I have done numerous samples by both methods and do not find any appreciable difference in the results. I thought it possible that the different treatment of non-tannin filtration might cause some difference in the non-tannin results of practically spent liquors and am not absolutely sure now that it does not, but all the work we have done so far points that way. I am indebted to Mr. J. R. Evans, an assistant in my laboratory, for some of the experimental work in this direction and the following table shows some of his results. These three liquors were taken from three vats of Rocker liquor and ought to show any existing difference due to the different non-tannin filtration.

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	No. 1.		No. 2.		No. 3.	
	Lab.	Ref.	Lab.	Ref.	Lab.	Ref.
Total Solids	1.96	1.91	2.91	2.86	2.20	2.18
Soluble Solids..	1.83	1.83	2.70	2.78	2.10	2.12
Non-tannin.....	1.43	1.37	1.58	1.50	1.47	1.44
Tannin.....	.40	.46	1.12	1.28	.63	.68
Insolubles.....	.13	.08	.21	.08	.10	.06

On the official samples the prescribed method gives lower reds and I expected this because of the dilution; but on the other hand, this method gave me lower total solids figures so I cannot argue that point on this particular example. I know it to be a fact, however, that dilution will diminish the reds found. With your dilution of liquors like Nos. 4, 5, 6, 50 grams of the average wet hide would not be sufficient. I find that for dirty liquors we must use an excess of hide as the insolubles clog the powder and prevent free absorption."

With regard to these criticisms, the proposed method does demand more attention, but seems likely to give more comparable results as the amounts dried are more uniform; further, as noted above, the experience of the chairman,—and this is confirmed by Mr. Cushing,—is that the amount of hide powder recommended is ample even for dirty liquors like Nos. 4, 5, and 6. The time of shaking, as practiced by Mr. Teas, the chairman, does not find sufficient to yield a uniform and complete absorption.

After carefully considering all the data at hand, the chairman is led to suggest the following as a provisional method for the analysis of spent liquors:

Make up solutions for analysis to contain .35 to .45 grams tannin per 100 cc., dilutions being made with water at 80° C. In each case evaporate such an aliquot portion as will give a residue for soluble solids of approximately from .7 to .8 grams. Dry all residues in 3" dishes by the official method.

To determine soluble solids, add 1 gram of kaolin to 75 cc. of liquor, diluted as above, stir and pour on single pleated filter. Collect filtrate and return through filter for one hour, keeping funnel well filled until well toward the end of the hour. Either pour out what remains in the filter or remove it by a pipette, let drain a few moments, add 75 cc. of fresh liquor and collect filtrate for drying as soon as clear. Keep funnel full.

Determine non-tannins by the official method. In cases where the original liquor contains less tannin than prescribed, it is per-

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missable to diminish proportionally the amount of hide powder used.

With regard to the other question outlined for the committee, namely, the effect on the analysis figures of the acid present in the liquor, Mr. Cushing reports as follows:

"About 10 liters of liquor of 1.003 gravity was prepared from fresh Hemlock Bark and immediately treated with Formalin to check acid formation. The liquor was divided into 10 parts and acidified with varying portions of Sulphuric, Lactic and Acetic Acids in the following way:—To about 950 cc. of the liquor varying quantities of approximately 50% acid solution were added, then sufficient water to make the total dilution 25 cc. in each case, and enough of the original liquor to make the volume 1 liter. After acidification the liquors were well shaken and allowed to stand until next day (20 hours) before analyzing.

The liquors acidified with Sulphuric Acid, Nos. 2, 3, 4, deposited considerable sediment on acidification, the solution more strongly acidified appearing quite clear at the top after standing some time.

The liquors acidified with Lactic Acid, Nos. 5, 6, 7, did not deposit an immediate sediment, but on standing some precipitation was noticed, depending upon the degree of acidity; No. 7, the strongest was considerably cleared at the top after settling out.

The liquors acidified with Acetic Acid, Nos. 8, 9, 10, showed some tendency to deposit sediment according to the degree of acidification, but did not show any very marked signs of clearing at the top on standing as was the case with the other acids. The temperature during analysis was 20° C. The percentages given in the following table were obtained from residues dried 8 hours at steam heat.

No.	Acid.	Grams Acid per Liter.	Per-centage Acid.	Total Solids.	Sol. Solids.	Ins.	Non. Tannins.	Tannins.
1	0	0	0	0.79%	0.52%	0.27%	0.21%	0.31%
2	Sulphuric	2.527	0.25	0.99	0.57	0.42	0.27	0.30
3	"	5.053	0.51	1.26	0.84	0.42	0.51	0.33
4	"	10.381	1.04	1.86	1.49	0.37	1.15	0.34
5	Lactic	0.725	0.07	0.86	0.60	0.26	0.24	0.36
6	"	3.645	0.36	1.07	0.71	0.36	0.35	0.36
7	"	7.290	0.73	1.32	0.75	0.47	0.50	0.25
8	Acetic	0.830	0.08	0.81	0.54	0.27	0.21	0.33
9	"	4.172	0.42	0.82	0.55	0.27	0.23	0.32
10	"	8.344	0.83	0.82	0.57	0.25	0.23	0.34

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The residues from the liquor acidified with sulphuric acid were in all cases considerably charred; they were very difficult to weigh with accuracy owing to the rapid increase of weight while weighing. On drying these residues for two hours more at steam heat it was found that eight hours was usually sufficient to bring them to constant weight. The same was found to be true of the liquors acidified with acetic acid. Quite serious differences were observed in the case of the liquors acidified with lactic acid upon further drying of the residues. Here the difficulty in drying was proportional to the degree of acidification. In the cases of Nos. 6 and 7 the soluble solids and non-tannin residues showed a continuous loss when dried at intervals which made a total of 32 hours, showing a loss in No. 6 of from 10 to 20 Mg. in the case of soluble solids residues between the residue dried for 8 hours and that dried for 32. The soluble solids and non-tannin residues of No. 7 showed a loss of about 20 Mg. between the first and last weighings for the former and from 90 Mg. to over one-tenth of a gram for the latter, and were still showing a loss of approximately 10 Mg. for each interval of 2 hours drying. The loss of weight for the soluble solids and non-tannins is shown by percentages calculated on the basis of the weighings last made, the former being .71%, the latter .36%, as compared with the first figures of .75% and .50%.

It was noticed that the filtered solution used for the determination of soluble solids in the case of Nos. 2, 3, 4, clouded considerably in 3 to 4 hours, while 5, 6, 7, 8, 9, 10, became cloudy in 7 to 8 hours, the temperature not falling below 20° C. The liquors were allowed to stand for 2 days, temperature not below 20°, and the soluble solids again determined with the following results:

1	2	3	4	5	6	7	8	9	10
0.49	0.48	0.78	1.36	0.53	0.62[1]	0.68[1]	0.49	0.47	0.49
....	....	....	....	....	0.59[2]	0.63[2]	....	....	....

[1] Dried in 8 hours.
[2] Dried in 32 hours.

It was noticed that these filtrates in turn became cloudy on standing some time at 20°, Nos. 2, 3, 4, being the first; so it is evident that the loss of soluble solids was continuing. From these results it appears that the Lactic Acid existing in liquors may introduce through its slow volatility appreciable errors in the determination, not so much perhaps in the percentage of tan-

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nin arrived at, if the conditions of drying are uniform, but in arriving at the true percentage of total and soluble solids and non-tannins. Acetic Acid, on the other hand, does not apparently interfere in this respect with the uniformity of results. Sulphuric Acid on account of its non-volatility and charring action on the residue makes results uncertain.

Acidification of the liquors appears to cause a gradual loss of soluble solids in all three cases, the extent of which can only be determined by taking up this point more fully.

The Effect of Neutralizing the Acid in a Tan Liquor.

About 4 liters of liquor of 1.003 gravity was prepared from fresh Hemlock Bark and treated with Formalin to check acid-formation. Three portions of the liquor were acidified to ½% with the Sulphuric, Lactic and Acetic Acid solutions already mentioned, the acid being added to about 950 cc. of the liquor in each case. After shaking and allowing to stand for a couple of hours the acid was exactly neutralized with a 33% solution of caustic soda, enough water added to make the dilution the same in all cases, the liquor thoroughly shaken and allowed to stand over night. One portion of the liquor was reserved untreated with the exception of adding sufficient water to compensate for the dilution by acid and alkali solutions in the other cases. In all cases a heavy sediment was formed on neutralizing, which settled, leaving the upper solution quite clear. The liquors were not cooled below 20° C. All residues dried 8 hours at steam heat.

No.	Acid.	% Salt in Sol. after Neutralizing.	Total Solids.	Sol. Sols.	Ins.	Non-Tan.	Tannins.
1	0	0	0.97	0.54	0.33	0.22	0.32
2	Sulphuric	0.77	1.64	1.09	.55	.97	.12
3	Lactic	0.73	1.60	1.14	.46	.96	.18
4	Acetic	0.66	1.53	1.06	.47	.92	.14

These results indicate that neutralization with caustic soda affects very seriously the insoluble and tannin figures, the results upon the acid liquors being more uniform in this respect. It is possible that neutralization with other agents may affect results less seriously. It is regretted that the writer through lack of time is compelled to leave these investigations in such an incomplete state."

These results are in line with those obtained by the chairman, though for lack of time his work was even less complete than that

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of Mr. Cushing. An oak liquor 19.7 Bk. and a Hemlock 18.3 Bk. were exactly neutralized with Barium Hydrate solution, then filtered till clear. To 400 cc. of clear solution in each case were added, respectively, 100 cc. of water; 100 cc. of Lactic Acid, containing 5 grams, of 72% Acid; 100 cc. of Acetic Acid, containing 10 grams, of 50% Acid. These solutions were then analyzed as usual. Portions of these liquors similarly acidified were neutralized by adding exactly the calculated amount of caustic soda and a total solids and non-tannin determination made in each case.

	Oak.			Hemlock.		
	No Acid.	5 gms. Lactic to 500 cc.	10 gms. Acetic to 500 cc.	No Acid.	5 gms. Lactic to 500 cc.	10 gms. Acetic to 500 cc.
Total Solids	3.547	4.178	3.754	3.594	4.162	3.774
Soluble Solids	3.525	4.155	3.721	3.600	4.159	3.785
Non-tannin.....	3.219	3.794	3.410	3.267	3.744	3.381
Tannin.....	.306	.361	.311	.333	.415	.404
Insolubles.....	.022	.023	.033	....	.003	....
	Neutralized with Caustic Soda.					
Total Solids		4.625	5.002		4.649	5.012
Non-tannin.....		4.336	4.697		4.309	4.664
Tannin.....		.289	.305		.340	.348

The results of Mr. Cushing and the chairman show that there is a certain amount of absorption of acid by the hide powder in the ordinary determination of the non-tannins in a sour liquor, but that only a small percentage of the amount of acid present is absorbed and the error introduced is in general not serious. Mr. Cushing's results show that any attempt to obviate this error by neutralizing the acid is probably merely calculated to make a bad matter worse. The results of the chairman contradict this in part, but he took no account of the precipitate form on adding the alkali and his non-tannin determinations were made immediately after the addition. Possibly under these conditions some method of neutralization might be effective, but until the subject has been more thoroughly investigated than this year's committee has been able to do, it seems wisest to make no attempt to correct for absorption of acid when making an analysis of a sour liquor.

FRITZ H. SMALL,
Chairman.

Following the findings of the Committee, the Referee included the proposed method under Study II (b) and requested analyses for comparison by the method employed in each individual laboratory, and later by yet another method which seemed well worthy of trial.

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STUDY II.

ANALYSIS OF LIQUORS.

(a) Complete analysis by method employed by each individual analyst accompanied by full and complete statement of all the details of such method.

(b) Determinations to be made according to the following method:

Make up solutions for analysis to contain .35 to .45 grams tannin per 100 cc., dilutions being made with water at 80° C. In each case evaporate such an aliquot portion as will give a residue for soluble solids of approximately from .7 to .8 grams.

To determine soluble solids add 1 gram of kaolin to 75 cc. of liquor, diluted as above, stir and pour on single-pleated filter. Collect filtrate and return through filter for 1 hour, keeping funnel well-filled until well towards the end of the hour. Either pour out what remains in the filter or remove it by a pipette, let drain a few moments, add 75 cc. of fresh liquor and collect filtrate for drying as soon as clear, keeping funnel full.

Determine non-tannins as in Study I (a). In cases where the original liquor contains less tannin than prescribed, it is permissible to diminish proportionately the amount of hide powder used. Evaporate same number of cc., as for total and soluble solids.

Temperature and method of drying pursued to be the same as given in Study I (a). Amount of wet hide and moisture content to be stated.

Proportions of spent liquors to be taken for analysis will be found on samples, with number of cc. to be pipetted.

(c) Dilution of Hemlock liquor for analysis, 80 cc. per liter.

Dilution of Oak liquor for analysis, 100 cc. per liter.

Filtrations for soluble solids to be conducted as per Study II (b), using temperature as per Study I (a). 11½ grams wet hide to be used per 200 cc. of the Hemlock liquor solution.

Six grams wet hide to be used per 200 cc. of the Oak liquor solution.

The results are tabulated below, eighteen analysts participating in the work.

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TABLE XIV.—STUDY II (a). SPENT HEMLOCK LIQUOR, LABORATORY METHOD.

Analyst	Total Solids	Soluble Solids	Insolubles	Non-Tannins	Tannins	Dilution cc. per Litre	Wet Hide Gms	Hide Powder Moisture %	Dry Hide Gms	Temperature Degrees C.	Notes
W. H. Teas, ...	5.71	5.45	.26	4.50	.95	N.T.—200	25	70.0	7.50	25	10 cc. undil. evap. for T. S. & S. S. N.T. shaken 3 minutes.
H. T. Wilson, ...	5.43	5.20	.23	4.27	.93	345	41	70.8	11.97	27	Dilution—Method B.
J. V. R. Evans, ...	5.73	5.61	.12	4.57	1.04	N.T.—200	25	70.0	7.50	25	10 cc. undil. evap. for T. S. & S. S. N.T. shaken 3 minutes.
W. K. Alsop and A. V. Hoppenstedt,...	5.62	5.43	.19	4.36	1.07	125	30	73.5	7.95	30	N.T. shaken 3 minutes.
Chas. Eachus, ...	5.63	5.42	.21	4.35	1.07	100	30	75.0	7.50	28	
M. F. Nichols,.....		5.34		4.36	.98	200	20	73.0	5.40	20—25	
F. W. Alden,..		5.48		4.73	.75	400					
G. A. Kerr, ...	5.76	5.46	.30	4.57	.89	200	30	68.5	9.45		
J. R. Mardick, ...		5.29		4.54	.75	333	20	71.3	5.74	22.5	
T. J. Mosser, ...	5.58	5.54	.04	4.46	1.08	200	30	80.0	6.00		N.T shaken 5 minutes.
F. H. Small, ...	5.42	5.21	.21	4.33	.88	345	43	70.4	12.75		Method B.
H. C. Reed, ...	5.65	5.40	.25	4.24	1.16	80	30	75.0	7.50	20—25	
C. W. Norris,.....	5.56	5.38	.18	4.19	1.19	143	40	69.0	12.4	30	
W. A. Fox, ...	5.66	5.30	.36	4.26	1.04	125	30	74.5	7.65		N.T shaken 3 minutes.
Average.....	5.62	5.39	.21	4.41	.98	214	30.3	72.4	8.41		
Maximum.....	5.76	5.61	.36	4.73	1.19	80	43.0	80.0	12.75		
Minimum	5.42	5.20	.04	4.19	.75	400	20.0	68.5	5.40		
Greatest Difference..	.34	.41	.32	.54	.41	320	23.0	11.5	7.35		

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TABLE XV.—STUDY II (b). SPENT HEMLOCK LIQUOR.

Dilution 345 cc. per Litre.

Analyst	Total solids	Soluble solids	Insolubles	Non-Tannins	Tannins	Wet Hide Gms	Hide Powder Moisture, %	Dry Hide Gms	Temperature Degrees C.	Notes
W. H. Teas,	5.65	5.45	.20	4.61	.84	50	70.00	15.00	25	
H. T. Wilson,	5.51	5.30	.21	4.31	.99	41	67.32	13.40	22	Laboratory Temperature, 29° C.
J. V. R. Evans,	5.66	5.41	.25	4.48	.93	50	70.00	15.00	25	
W. K. Alsop and A. W. Hoppenstedt,	5.59	5.38	.21	4.36	1.02	50	73.50	13.25	30	
Chas. Eachus,	5.54	5.36	.18	4.50	.86	50	75.00	12.50		
M. F. Nichols,	5.69	5.49	.20	4.49	1.00	30	73.00	8.10	20—25	
E. G. Wilmer,	5.56	5.33	.23	4.37	.96	45.5	72.24	12.63		
F. P. Veitch,	5.34	5.14	.20	4.16	.98	50	78.00	11.00	28—32	
H. H. Hurt,	5.53	5.34	.19	4.29	1.05	50	76.40	11.80	28—32	
C. R. Delaney,	5.71	5.47	.24	4.41	1.06	35	66.40	11.76	25.5	
G. D. McLaughlin,	5.45	5.26	.19	4.30	.96	41	70.79	11.98		
G. A. Kerr,	5.63	5.54	.09	4.59	.95	40	65.00	14.00		
F. H. Small,	5.42	5.21	.21	4.33	.88	43	70.40	12.75	23	
F. W. Alden,		5.28		4.55	.73					
H. C. Reed,	5.56	5.32	.24	4.38	.94	46	73.56	12.16	22	
C. W. Norris,	5.60	5.38	.22	4.35	1.03	49	71.00	14.21	28—31	
F. R. Mosbaugh,	5.56	5.36	.20	4.39	.97	40	69.20	12.32		
W. A. Fox,	5.67	5.34	.33	4.37	.97	50	74.50	12.75		
Average,	5.57	5.35	.21	4.40	.95	44.7	71.55	12.62		
Maximum,	5.71	5.54	.33	4.61	1.06	50.0	78.00	15.00		
Minimum,	5.34	5.14	.09	4.16	.73	30.0	65.00	8.10		
Greatest Difference,	.37	.40	.24	.45	.33	20.0	13.00	6.90		

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TABLE XVI.—STUDY II (c). SPENT HEMLOCK LIQUOR.

Dilution 80 cc. per Litre.—Wet Hide 11½ Gms., per 200 cc. Solution.

Analyst	Total Solids	Soluble Solids	Insolubles	Non-Tannins	Tannins	Hide Powder Moisture %	Dry Hide Gms	Temperature Degrees C.	Notes
H. T. Wilson,	5.46	5.22	.24	4.20	1.02	69.6	3.50	28—32	
W. K. Alsop and A. W. Hoppenstedt,	5.59	5.45	.14	4.41	1.04	73.5	3.05	30	
Chas. Eachus,	5.58	5.33	.25	4.32	1.01	75.0	2.88	28	
M. F. Nichols,		5.40		4.42	.98	75.0	2.98	20—26	
E. G. Wilmer,		5.45		4.40	1.05	70.1	3.44	25	
F. W. Alden,		5.33		4.32	1.01	70.1	3.44	20 +	
C. R. Delaney,	5.65	5.37	.28	4.28	1.09	67.2	3.77	20—27	
F. H. Small,	5.42	5.22	.20	4.24	.98	70.4	3.40		
H. C. Reed,	5.65	5.40	.25	4.39	1.01	75.0	2.88	20—25	
F. R. Mosbaugh,	5.64	5.41	.23	4.35	1.06	69.2	3.54	20—23	N.T.—using 3 gms. D.C.H.—4.43.
W. A. Fox,	5.69	5.31	.38	4.39	.92	74.5	2.93		
Average,	5.59	5.35	.246	4.34	1.016	71.8	3.25		
Maximum,	5.69	5.45	.38	4.42	1.09	75.0	3.77		
Minimum,	5.42	5.22	.14	4.20	.92	67.2	2.88		
Greatest Difference,	.27	.23	.24	.22	.17	7.8	.89		

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TABLE XVII.—STUDY II (a). SPENT OAK LIQUOR.

Laboratory Method.

Analyst	Total Solids	Soluble Solids	Insolubles	Non-Tannins	Tannins	Dilution cc. per Litre	Wet Hide Gms	Hide Powd. Moisture %	Dry Hide Gms	Temperature Degree C.	Notes
W. H. Teas,	5.03	4.84	.19	4.21	.63	N.T.—200	25	70.0	7.50	25	10 cc. undil. evap. for T.S. & S.S. N.T. shaken 3 minutes.
H. T. Wilson,	4.79	4.59	.20	3.99	.60	N.T.—375	41	70.8	11.97	27	T.S. & S.S. dil. 300 cc. to 1 litre.
J. V. Evans,	5.04	4.95	.09	4.22	.73	N.T.—200	25	70.0	7.50	25	10 cc. undil. evap. for T.S. & S.S. N.T. shaken 3 minutes.
W. K. Alsop and A. W. Hoppenstedt,	4.91	4.78	.13	4.09	.69	200	30	73.5	7.95	30	N.T. shaken 3 minutes.
Chas. Eachus.	5.00	4.82	.18	4.05	.77	100	30	75.0	7.50	28	
M. F. Nichols,		4.73		4.09	.64	200	20	73.0	5.40	20—25	
F. W. Alden,[1]		4.45		4.38	.07	500					
G. A. Kerr,	4.89	4.74	.15	4.07	.67	250	40	65.0	14.00		
J. R. Mardick,		4.62		4.13	.49	333	20	71.3	5.74	22.5	
T. J. Mosser,	4.98	4.86	.12	4.20	.66	200	30	80.0	6.00		N.T. shaken 3 minutes.
F. H. Small,	4.90	4.72	.18	4.37	.35	1000	46.4	72.9	12.57		Method B.
H. C. Reed,	4.89	4.72	.17	3.96	.76	100	30	75.0	7.50	20—25	
C. W. Norris,	4.89	4.71	.18	4.11	.60	167	15	71.6	4.25	27	
W. A. Fox,	4.90	4.74	.16	4.05	.69	200	30	74.5	7.65		N.T. shaken 3 minutes.
Average.	4.93	4.76	.16	4.12	.64	287.5	29.4	72.5	8.12		
Maximum,	5.04	4.95	.20	4.37	.77	100	46.4	80.0	14.00		
Minimum,	4.79	4.59	.09	3.96	.35	1000	15.0	65.0	4.25		
Greatest Difference,	.25	.36	.11	.41	.42	900	31.4	15.0	9.75		

[1]Not included in average.

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TABLE XVI.—STUDY II (c). SPENT HEMLOCK LIQUOR.

Dilution 80 cc. per Litre.—Wet Hide 11½ Gms., per 200 cc. Solution.

Analyst	Total Solids	Soluble Solids	Insolubles	Non-Tannins	Tannins	Hide Powder Moisture %	Dry Hide Gms	Temperature Degrees C.	Notes
H. T. Wilson,	5.46	5.22	.24	4.20	1.02	69.6	3.50	28—32	
W. K. Alsop and A. W. Hoppenstedt,	5.59	5.45	.14	4.41	1.04	73.5	3.05	30	
Chas. Eachus,	5.58	5.33	.25	4.32	1.01	75.0	2.88	28	
M. F. Nichols,		5.40		4.42	.98	75.0	2.98	20—26	
E. G. Wilmer,		5.45		4.40	1.05	70.1	3.44	25	
F. W. Alden,		5.33		4.32	1.01	70.1	3.44	20 +	
C. R. Delaney,	5.65	5.37	.28	4.28	1.09	67.2	3.77	20—27	
F. H. Small,	5.42	5.22	.20	4.24	.98	70.4	3.40		
H. C. Reed,	5.65	5.40	.25	4.39	1.01	75.0	2.88	20—25	
F. R. Mosbaugh,	5.64	5.41	.23	4.35	1.06	69.2	3.54	20—23	N.T.—using 3 gms. D.C.H.—4.43.
W. A. Fox,	5.69	5.31	.38	4.39	.92	74.5	2.93		
Average,	5.59	5.35	.246	4.34	1.016	71.8	3.25		
Maximum,	5.69	5.45	.38	4.42	1.09	75.0	3.77		
Minimum,	5.42	5.22	.14	4.20	.92	67.2	2.88		
Greatest Difference,	.27	.23	.24	.22	.17	7.8	.89		

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TABLE XVII.—STUDY II (a). SPENT OAK LIQUOR.

Laboratory Method.

Analyst	Total Solids	Soluble Solids	Insolubles	Non-Tannins	Tannins	Dilution cc. per Litre	Wet Hide Gms	Hide Powd. Moisture %	Dry Hide Gms	Temperature Degree C.	Notes
W. H. Teas,	5.03	4.84	.19	4.21	.63	N.T.—200	25	70.0	7.50	25	10 cc. undil. evap. for T.S. & S.S. N.T. shaken 3 minutes.
H. T. Wilson,	4.79	4.59	.20	3.99	.60	N.T.—375	41	70.8	11.97	27	T.S. & S.S. dil. 300 cc. to 1 litre.
J. V. Evans,	5.04	4.95	.09	4.22	.73	N.T.—200	25	70.0	7.50	25	10 cc. undil. evap. for T.S. & S.S. N.T. shaken 3 minutes.
W. K. Alsop and A. W. Hoppenstedt,	4.91	4.78	.13	4.09	.69	200	30	73.5	7.95	30	N.T. shaken 3 minutes.
Chas. Eachus.	5.00	4.82	.18	4.05	.77	100	30	75.0	7.50	28	
M. F. Nichols,		4.73		4.09	.64	200	20	73.0	5.40	20—25	
F. W. Alden,[1]		4.45		4.38	.07	500					
G. A. Kerr,	4.89	4.74	.15	4.07	.67	250	40	65.0	14.00		
J. R. Mardick,		4.62		4.13	.49	333	20	71.3	5.74	22.5	
T. J. Mosser,	4.98	4.86	.12	4.20	.66	200	30	80.0	6.00		N.T. shaken 3 minutes.
F. H. Small,	4.90	4.72	.18	4.37	.35	1000	46.4	72.9	12.57		Method B.
H. C. Reed,	4.89	4.72	.17	3.96	.76	100	30	75.0	7.50	20—25	
C. W. Norris,	4.89	4.71	.18	4.11	.60	167	15	71.6	4.25	27	
W. A. Fox,	4.90	4.74	.16	4.05	.69	200	30	74.5	7.65		N.T. shaken 3 minutes.
Average.	4.93	4.76	.16	4.12	.64	287.5	29.4	72.5	8.12		
Maximum,	5.04	4.95	.20	4.37	.77	100	46.4	80.0	14.00		
Minimum,	4.79	4.59	.09	3.96	.35	1000	15.0	65.0	4.25		
Greatest Difference,	.25	.36	.11	.41	.42	900	31.4	15.0	9.75		

[1]Not included in average.

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TABLE XVIII.—STUDY II (b). SPENT OAK LIQUOR.

Undiluted.

Analyst	Total Solids	Soluble Solids	Insolubles	Non-Tannins	Tannins	Wet Hide Gms	Hide Powder Moisture %	Dry Hide Gms	Temperature Degree C.	Notes
W. H. Teas,	4.94	4.69	.25	4.42[1]	.27	50	70.00	15.00	25	
H. T. Wilson,	4.91	4.72	.19	4.16	.56	41	67.32	13.40	22	N.T. dil. 100—200
J. V. R. Evans,	4.96	4.72	.24	4.48[1]	.24	50	70.00	15.00	25	
W. K. Alsop and A. W. Hoppenstedt	4.93	4.85	.08	4.49[1]	.36	50	73.50	13.25	30	
Chas. Eachus,	4.93	4.86	.07	4.20	.66	50	75.00	12.50	28	
M. F. Nichols,	4.97	4.83	.14	4.38	.45	30	73.00	8.00	20—25	
E. G. Wilmer,	4.74	4.67	.07	4.40	.27	45.5	72.24	12.63		
F. W. Alden,		4.76		4.50	.26					
C. R. Delaney,	4.99	4.76	.23	4.47	.29	35	66.40	11.76	25.5	
G. D. McLaughlin,	4.81	4.58	.23	4.03	.55	41	70.79	11.98		
G. A. Kerr,	4.84	4.70	.14	4.14	.56	50	70.50	14.75		
F. P. Veitch,	4.93	4.71	.22	4.42	.29	50	78.00	11.00	28—32	
H. H. Hurt,	4.91	4.73	.18	4.39	.34	50	76.40	11.80	28—32	
F. H. Small,	4.90	4.72	.18	4.37	.35	46.4	72.90	12.57	23	
H. C. Reed,	4.95	4.78	.17	4.36	.42	46	73.56	12.16	22	
C. W. Norris,	4.93	4.76	.17	4.22	.54	45	71.00	13.05	28—31	
F. R. Mosbaugh,	5.02	4.81	.21	4.41	.40	40	69.20	12.32		
W. A. Fox,	4.96	4.80	.16	4.38[1]	.42	50	74.50	12.75		
Average,	4 92	4.75	.17	4.35	.40	45.3	72.02	12.59		
Maximum,	5.02	4.86	.25	4.50	.66	50.0	78.00	15.00		
Minimum,	4.74	4.58	.07	4.03	.24	30.0	66.40	8.00		
Greatest Difference	.28	.28	.18	.47	.42	20.0	11.60	7.00		

[1]Not clear.

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TABLE XIX.—Study II (c). SPENT OAK LIQUOR.

Dilution 100 cc. per Litre. Wet Hide Powder 6 gms. per 200 cc. Solution.

Analyst	Total Solids	Soluble Solids	Insolubles	Non-Tannins	Tannins	Hide Powder Moisture %	Dry Hide Gms	Temperature Degree C.	Notes
W. H. Teas,	4.93	4.70	.23	4.27	.43	80.0	1.20	25	
H. T. Wilson,	4.80	4.60	.20	4.10	.50	69.6	1.82	28—32	
J. V. R. Evans,	4.88	4.70	.18	4.27	.43	70.0	1.80	25	
W. H. Alsop and A.W. Hoppenstedt,	4.90	4.76	.14	4.25	.51	73.6	1.59	30	
Chas. Eschus,	4.90	4.77	.13	4.20	.57	75.0	1.50	28	
M. F. Nicholls,		4.89		4.17	.72	75.0	1.50	20—26	
E. G. Wimer,		4.62		4.15	.47	70.1	1.79	25	
F. W. Alden,		4.55		4.12	.43	70.1	1.79	20 +	
C. R. Delaney,	4.96	4.72	.24	4.21	.51	67.2	1.97	20 +	
F. P. Veitch,	4.76	4.70	.06	4.12	.58	72.8	1.63	28—32	
F. H. Small,	4.84	4.66	.18	4.19	.47	72.9	1.63		
H. C. Reed,	4.89	4.72	.17	4.25	.47	75.0	1.50	22	N.T. using 1.5 gms. D.C.H.—4.31%
W. A. Mosbaugh,	4.91	4.73	.18	4.22	.51	69.2	1.85	20—23	
W. A. Fox,	4.89	4.74	.15	4.27	.47	74.5	1.53		
Average,	4.88	4.70	.17	4.20	.505	72.5	1.65		
Maximum,	4.96	4.89	.24	4.27	.72	80.0	1.97		
Minimum,	4.76	4.55	.06	4.10	.43	67.2	1.20		
Greatest Difference,	.20	.34	.18	.17	.29	12.8	.77		

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Collaborators' Notes Relative to Study II.

W. H. Teas.

Mr. Teas submits figures on Referee's amended method (c), showing non-tannin results with various amounts of hide powder,

Spent Oak Liquor.	
Wet Hide. Gms.	Non-tannins.
10	4.21
15	4.18
20	4.08
25	4.06

He says:

"Our laboratory method for liquors consists in drying 10 cc. of the shaken liquor for total solids, adding 2 grams kaolin to 200 cc. liquor, stir and throw on Prat-Dumas double filter, allowing filtration to proceed 30 minutes, then the filtrate is collected and 10 cc. taken for evaporation. For non-tannins, I dilute 50, 100 or 200 cc. of the liquor to 500 cc., according to the strength of the liquor, use 200 cc. of the dilution and shake with hide powder 3 minutes. I use from 15 to 45 grams of wet hide powder according to the strength and character of the liquor. After shaking, add kaolin, stir and filter through double Prat-Dumas, collecting 100 cc., after the filtrate runs clear.

I use eight, numbered pipettes and hold up the pipette until it is empty, and then allow it to drain while the other seven are in use and while the non-tannin dilutions are being made, about 10 minutes. In collecting the soluble solids the same pipette is used for each liquor as was used in collecting the total solids of that liquor and the same time is allowed for draining.

For liquors above 20° Bark. I dilute for non-tannins, 50 cc. to 500 cc., from 10° to 20°, 100 cc. to 500 cc. below 10°, 200 cc. to 500 cc. For liquors of the first dilution I use from 30 to 45 grams wet hide; of the second dilution, from 20 to 35 grams, and of the last dilution from 15 to 25 grams wet hide, for 200 cc. of the liquor. The amount used depends on the strength and nature of the liquor.

The samples were worked by the Referee's method (b), and the laboratory method (a) on the same day. The determination by the Referee's amended method was made at another time and with hide powder, chromed by two additions of chrome.

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I could not obtain a clear non-tannin filtrate on the oak liquor, with the amount of hide powder named in the Referee's directions.

I do not think that there should be much difference in the total solids and soluble solids results by the three methods. The undiluted method has the advantage of testing the liquors just as they are in the tannery, and it is a time-saver. The chance of error in pipetting such small amount is reduced to a minimum by allowing a uniform draining time, and the use of tested, numbered pipettes. The dried residue is just as frequently thinner than the residue of a limited total solids dilution, as it is thicker. The first stage of evaporation will subject a diluted solution to sustained heat and the residue will then be where the undiluted solution started. The undiluted residue will have more drying after evaporation, but I do not think there is much chance of oxidation in the dry,hard residue.

In laboratories where large numbers of liquors are analyzed it would be impracticable to figure out the dilutions, and amounts to be evaporated for each liquor.

All three methods call for non-tannin determinations on a diluted liquor. (There may be occasional exceptions to this in method (b)). On the non-tannin determinations, therefore, there should not be any variation, except that due to the different amounts of hide powder used.

The variations in the non-tannin figures (aside from the laboratory error) on these samples is probably due to the different amounts of powder used. For 200 cc. of the hemlock analysis solution containing, say .621 gram tannin, the Referee's method calls for 12-13 grams dry hide; on 200 cc. of the oak analysis solution containing, say .9 (1.06) gram tannin, the same amount is called for; in the amended method, for a quantity representing, say .09 (.106) gram tannin, about 1½ grams dry hide is used. The above tannin amounts are figured on the basis of average of results by the two methods; the figures in parentheses represent amounts figured on basis of average of results by Referee's amended method and the laboratory method, which average, I believe, to be nearer the actual tannin contents of the liquor.

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The following table shows the amounts of hide powder recommended, and their relation to the tannin contents of the liquor:

	Dry Hide Recommended	Tannin in 200 cc used	Amount Tannin for 1 gr. H. P.
Hemlock Liquor	12½ gr.	.621 gr.	.050 gr.
Oak Liquor (b)	12½ gr.	(1.06)	.085
		.90	.072
Oak Liquor	1½ gr.	(.106)	.0706
(Amended Method)		.09	.060

On the first two liquors the hide powder actually used was dryer than expected, so that the following larger amounts were made:

	Dry Hide	Tannin in 200 cc.	Tannin for 1 gr. H. P.
Hemlock Liquor:			
Referee (b)	15 gr.	.621 gr.	.041 gr.
Laboratory Method	7½	.36	.048
Oak Liquor:			
Referee (b)	15	(1.06)	.070
		.9	.060
Referee (Amended)	1.2	(.106)	.088
		.09	.075
Laboratory Method	7.5	(.212)	.028
		.18	.024

On a Quebracho extract containing 38% tannin, the official method requires 10 grams dilution, and 12½ grams dry hide for 200 cc., or 1 gram dry hide for .061 gram tannin. This extract liquor would contain non-tannins to the extent of about 25% of its tannin content which would not clog the powder and interfere with the absorption of the tannin. While it might seem advisable to decrease the proportion of hide powder for use in a liquor where there is such a large amount of non-tannins that absorption (of N. T.) might take place, yet I believe that the amount of hide powder used for liquors should be greater in proportion to the tannin than that used on extracts. Especially, should this be the case with respect to sapped oak liquors, in which the purity is low, and where the excess of non-tannins and the presence of considerable Gallic acid interfere with the tannin absorption, and renders difficult a clear filtrate with the use of a small amount of hide powder. In the case of new liquors the effect of different amounts of powder is not so great; on spent hemlock liquors it is more noticeable, and is greatest with spent oak liquors.

While running through the amended method, I made additional non-tannin determinations using larger amounts of hide powder. These results show the effect of the additional powder used.

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H. T. Wilson.

"Our laboratory method is the same as outlined in Study II (b), except that soluble solids is made as per official method for extract, etc."

W. K. Alsop and A. W. Hoppenstedt.

"Thirteen grams of dry hide were used for analyses under (b) as no amount was specified, and we had not had the advantage of having analyzed the liquors before. All the filtrates obtained during these analyses were clear, except those from Oak (b). They filtered very slowly, the non-tannin filtrate, especially, was turbid even after returning to the filter a number of times. The low tannin obtained by this method we consider to be due to this cause. The solutions under (a) were made by diluting with cold water, shaken, 100 cc. evaporated for solids. For soluble solids 150 cc. was filtered through 18 cm. paper, allowing all to filter through; 150 cc. of fresh solution was put in filter and 100 cc. of filtrate evaporated. For non-tannins 200 cc. was shaken three minutes with 30 grams of wet hide containing 73.50% water, thrown on filter containing 2 grams kaolin and 100 cc. clear filtrate evaporated.

Analyses under (c) were made in accordance with your instructions contained in your letter of August 13th. This method we consider preferable to the one under Study II (b), but do not consider it advisable to dilute with water at 80° C., or to make the solutions so weak.

We dilute liquors so as to contain approximately .6 gram per 100 cc., except in the case of very weak liquors when we make the solutions more concentrated. Thirty grams of wet hide are used for all determinations, and shaken for 3 minutes with 200 cc. of solution.

As we understand the method under Study II (b), it is to dilute the liquor so as to contain .35 to .45 gram tannin per 100 cc., and to evaporate such an amount of the solution as will give a residue of .7 gram. There are some objections to this method as an *Official* one. As we understand it, an Official method should be one that is generally applicable, to Trades laboratories as well as to laboratories of this character.

It would not be advisable to use a method such as this in the

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laboratory here, unless there are very decided advantages in it, which we are unable to see, to offset the disadvantages which are very apparent; and, in our opinion, it is only in a laboratory of this character where the tannin content of the various liquors can be approximated, that such a method could be used to advantage, if it were advisable.

Dilution with water at 80° C. we consider to be of very doubtful advantage and unnecessarily increasing the time required to make the analyses. The evaporation of various quantities of solution for residues introduces many chances of errors, makes the method more complicated and lengthens the time required. It possesses no advantage that we can see.

Liquors which especially require to be diluted to obtain satisfactory filtrates (such as the sample of Oak liquor) are the ones which have no dilution or at best the least. The question of time figures here also, as liquors of this character often filter very slowly.

We would suggest that the method should call for a dilution to give a certain amount of solids per 100 cc. This can be easily approximated from the Barkometer degree. .06 or .07 gram per 100 cc., and 30 or 40 grams of wet hide containing from 70 to 75% of water per 200 cc. of solution would perhaps be satisfactory.

One of two courses could be followed for the non-tannins; either use the same amount of hide for all solutions regardless of the tannin content, or if it be deemed advisable to decrease the amount of hide for weaker liquors it could be done at least as satisfactorily as by the other method, and in the way you suggested for samples (c) and as we did for samples (a)."

Chas. Eachus.

"By the laboratory method I filtered for soluble solids by adding 5 grams kaolin to 150 cc. liquor, stirring, and decanting on pleated filter. When 100 cc. was through, I poured filter full, and collected clear filtrate for soluble solids."

M. F. Nichols.

"Filtration is more rapid in oak liquor on account of dilution, and proportion of dry hide powder to tannin is more correct than in Study II (b) per each 200 cc. of solution."

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E. G. Wilmer.

"The method used by us is practically the same as that described under Study II (b). The only material difference is that our spent liquors are diluted up so as to enable us to evaporate 100 cc., instead of 15 or 40 cc."

G. A. Kerr.

"In our laboratory practice, we dilute all liquors, except those from tail handlers, and those we know to be quite low in tannin, to a strength compatible, as nearly as possible, with the dilution of extracts, and complete the analysis as per Official method. In the case of tail handler liquors and those which, in our judgment, are low in tannin, we reduce the quantity of hide powder in proportion."

T. J. Mosser.

"The laboratory method pursued by Mr. Mosser is similar to that used at the laboratory of the United States Leather Company."

J. R. Mardick.

"The liquors were diluted with water at 30° C., left over night and analyzed next morning. We do not like the idea of diluting liquors with water at 80° C. Nothing should be done to tamper with the natural conditions of liquors."

C. W. Norris.

"I dilute the liquors according to the degree, so as to have .7 to .8 of a gram of soluble solids, and vary the weight of hide so as to have about 12 grams for .3 to .4 of a gram (in that proportion) of tannin per 100 cc.

Dilutions are made with water at room temperature.

For evaporations 100 cc. are used for each determination.

I allow about 225 cc. of liquor to run through the filter before collecting the 100 cc. for soluble solids. As a rule, I find no difficulty in having rapid filtering so it is an easy matter to discard 225 cc. before collecting the 100 cc. for soluble solids."

Under Study II (b), Mr. Norris says:

"It is my opinion that it is not necessary to make the dilutions with water at 80° C.

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It would often save the time of standing over night if the dilution could be made with water at room temperature.

So along with the work as requested I have made dilutions upon the spent Bark. liquor both ways and run the analyses side by side."

Mr. Norris' results show identical soluble solids by diluting with water at 80° C., and with water at room temperature, and a difference of but .01% in non-tannins.

He accounts for the high non-tannin in the case of the oak liquor, undiluted, by the fact that the tannin per 100 cc. amounted to .52 gram higher than directions called for.

W. A. Fox.

"All the filtrates were clear, except those from Oak (b). Analyses (a) were made according to a laboratory method similar to Messrs. Alsop's and Hoppenstedt's method."

The returns from the various collaborators and their remarks relative to Method B, would seem to indicate that the method as suggested by the Chairman of the Committee is not one that would give, in the hands of various analysts, sufficiently concordant results to warrant its adoption as a provisional method. Moreover, Method C, as shown by the tables, produced an excellent concordance, and would indicate that weaker dilutions than those recommended in the Liquor Committee's report, with a reduction in hide powder, corresponding to the reduction in tannin, would better answer the purpose for a provisional method.

It is true that by using solutions diluted to give an approximately fixed residue for solids we are departing from the rule which specifies that extracts shall be diluted so as to contain between .35-.45 gram tannin per 100 cc., but it is equally true that if we dilute liquors to contain per 100 cc., the limits of tannin stated, we are obliged to depart from the rule which calls for the evaporation of 100 cc. in all cases.

It may be that the wider variations in results by Method B, were due in part to the unfamiliarity of the analysts with the technicalities of the method. But if this is the case it is not very plainly evinced in the total and soluble solids, which would eliminate as the source of error the variation in the system of pipetting, although the differences in these items are greater than by

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Method C. It is in the non-tannin figure, apparently, that Method B shows greatest weakness, and some of the collaborators have complained that they were unable to get clear non-tannin filtrates with the oak liquor. The Referee had, himself, no little trouble in this respect, and it was only after many returnings of the filtrate that a clear solution was obtained. With Method C, the Referee experienced no difficulty with the non-tannin determination, and nothing is said in the several reports that would lead him to believe that other analysts had difficulty.

Theoretically, the employment of a more diluted solution for analysis, with a proportionally decreased quantity of hide powder, should result in changes only in the soluble solids and insolubles items, where the influence of the dilution might produce lower insolubles, but as a matter of fact the average insolubles in both the Oak and Hemlock liquors is a trifle higher by Method C than by Method B.

The Referee fails to see the advantage of the method used by Mr. Teas (see Mr. Teas' report, Study II). If a dilution has to be made for the non-tannin estimation it might just as well be made for the entire analysis, as there would be no saving in time, and the time, labor and accuracy consumed in the pipetting and draining of 10 cc., would appear to equal, if not exceed, that for pipetting 100 cc., in the usual manner.

The Referee does not consider it advisable to dilute the liquors with hot water, as there is no very marked difference in result when this is done, compared with cold dilution, and considerable times is lost thereby. Moreover, tannery liquors are not subjected to a similar elevation of temperature.

All things considered, therefore, the Referee is inclined to advocate a method calling for a certain solids content, based upon the Barkometer degree and the character of liquor, and the use of an amount of hide powder to a degree corresponding with that called for by the official method in extract analysis. He therefore recommends the following provisional method.

Provisional Method for Liquor Analysis.

Liquors must be diluted for analysis so as to give as nearly as possible .7 grams of solids per 100 cc.

Total Solids to be determined as in extract analysis.

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Soluble Solids determination to be made in the same manner as with solutions of extracts.

Non-tannin determinations must be made by shaking 200 cc. of solution with a proportion of wet chromed hide powder containing 70 to 75% moisture, according to the following table:

Tannin range per 100 cc.	Dry Hide per 200 cc. Solution.
.35 — .45 gms.	10 — 12 gms.
.25 — .35 "	7 — 10 "
.15 — .25 "	4¼ — 7 "
. 0 — .15 "	0 — 4¼ "

100 cc. must be evaporated in each case and the method of evaporation and drying pursued to be the same as for extract analysis.

The proportion of hide powder to tannin is purposely made less than in the case of extract analysis owing to the fact that the acidity of liquors causes the hide to absorb the tannin more readily.

The result of the investigation made by the "Liquor" Committee upon the influence of the acids of liquors upon the analysis would indicate that these have but slight effect upon the tannin item, and that an attempt to neutralize the acidity previous to analysis would be liable to lead to greater error than when analyzed in their natural condition.

As the Chairman states, however, the subject has received but partial investigation, and the Referee recommends that it be gone into more fully next year.

STUDY III.

EXTRACTION OF TANNING MATERIALS.

The following suggestions were made to the Chairman of the "Extraction" Committee, Mr. Teas, as to line of work that might profitably be pursued:

EXTRACTION.

Suggested Lines of Investigation.

1. Extraction of Tannin materials in Soxhlet, arranged so that the earlier fractions of extractive may be collected separately, removed, and the extraction continued with the remaining portion in the usual manner.

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2. Comparison showing results obtained by extraction in various forms of extractors. It is recommended that the form suggested by Mr. Kerr in his report to the A. O. A. C. last year, be given a thorough trial.

3. The influence of laboratory extraction upon the Soluble Solids and Non-tannins, especially from the prolonged boiling in the Soxhlet extractor.

4. In connection with 3, the effect of the extraction upon spent tan, to prove whether true results are at present returned by the analysis.

5. Influence of time of extraction, amount of material extracted and proportion of water used for extraction, as shown by the analysis.

The report of the committee came into the possession of the Referee too late to enable him to incorporate the conclusions arrived at in the scheme for collaborative work.

Committee Report on Extraction.

Instructions Given by Chairman to Committee.

For the work on the extraction of tanning materials the following samples have been sent you:

New and Spent Hemlock Bark.
New and Spent Chestnut Wood.

Please extract a portion of these samples according to the following directions as to type of extractor, time of extraction, amount to be used for extraction, etc.:

(1) **Soxhlet extractor, 10 hours extraction.**
(2) **Percolation method described by Proctor.**
(3) **Method described by G. A. Kerr, p. 17, Report, 1904.**
(4) **Soxhlet extractor, allowing of removal of first two cups.**

Use 25 grams (absolutely dry) of				**new bark**	**With one liter of distilled water.**
" 80	"	"	"	**spent bark**	
" 40	"	"	"	**new wood**	
" 80	"	"	"	**spent wood**	

Analyze extracted matter at 20° C., as follows:

Total Solids.—Shake in a liter cylinder, allow to stand ½ hour, remove 100 cc. for evaporation.

Soluble Solids.—Add 5 grams kaolin to 150 cc. of the liquor, stir, throw on filter, allow to filter for ½ hour; throw away this filtrate, add fresh liquor to the filter, and collect 100 cc. of this filtrate for evaporation.

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Non-tannins.—Add pressed chromed hide powder to 200 cc. of the liquor, shake four minutes; add 5 grams kaolin, stir, throw on double filter paper, and collect 100 cc. of the filtrate as soon as it runs clear. For the new bark and wood use 45 grams hide (70-75% moisture). For the spent bark and wood use 30 gr. hide (70-75% moisture).

A more complete study of the comparative results of *different* methods of extraction upon the *same* bark under otherwise precisely similar conditions as to amount taken, time, etc., would be greatly appreciated.

On other barks and woods convenient to you, I should like you to do all the work that you feel inclined to do towards enlightenment on the following points: The various influences of amount of sample taken, duration and temperature of extraction, and the preparation of the sample upon the analysis.

I should also like opinions on the minimum amount of material necessary for moisture determinations.

In reporting results please state method and duration of drying residues.

Committee Report.

The comparative work of this committee consisted in the analysis of new and spent hemlock bark, and new and spent chestnut wood, after extraction by the different laboratory methods of extraction most commonly in use.

Four methods of extraction were specified in the chairman's directions: (1) The ordinary Soxhlet; (2) by the ordinary Soxhlet allowing of the removal of the first or strong portions of the extraction so that this would not be subjected to continuous boiling; (3) by Procter's method, and (4) by a percolation method suggested and used by Mr. G. A. Kerr. The Soxhlet and Procter methods are familiar to all; the regular Soxhlet apparatus is capable of arrangement in different ways in order to effect the removal of the first portions of the extraction. Mr. Kerr's method consists in allowing condensed steam at the boiling temperature to slowly percolate a column of the material to be extracted. This is effected by leading steam through an air condenser, which delivers the condensation to the top of the vertical cylinder containing the material to be extracted, and the soluble extractive is

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continuously delivered through a tube in the bottom of the cylinder.

Results of the different methods of extraction are shown in Tables 1, 2, 3 and 4. It is to be regretted that all the members did not extract all the samples by the four methods so that the averages could be reckoned on the maximum number of determinations. Figured on the averages of the analyses made, Mr. Kerr's method gives the highest results on two of the samples, and the Soxhlet method in which the first portions of the extraction were removed gave maximum results on the other two. It is interesting, although, because of the incompleteness of the tables, hardly significant, that the Kerr apparatus gave highest results on the new samples, and the modified Soxhlet on the spent samples. The results by the ordinary Soxhlet extractor were not very much lower than the maximum for each sample.

From previous experience not quite so favorable as the results here shown, I am of the opinion that the Procter method should be eliminated as a factor in the extraction of *spent* tanning materials.

On the different methods of extraction, Mr. Eachus writes: "I have found that the old Soxhlet method sometimes gives a great deal more reds and less tannin in barks than at other times. The Soxhlet method with removal of first portions of extraction seems to give the highest tannin..... I could not extract the spent samples by Procter's method so report no results that way. Kerr's method seems to be satsifactory, except that it is hard to regulate; in Procter's and Kerr's methods I had to collect 2,000 cc. solution in order to extract thoroughly..... A modification of Kerr's percolator method of extraction ought, in my opinion, to be the best extractor."

Mr. Kerr writes as follows: "While we can scarcely go so far as to assert the relative value of the two methods employed, with the scanty number of analyses made, I am disposed to think, that, under the proper conditions, the percolator system involves the better principle of extraction.

"You will note that in every case but one, the purity or ratio of tannin to soluble solids, is higher than in the Soxhlet, which would indicate some inversion of tannin, due to prolonged boiling over a direct flame.

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"I would also call your attention to the percolator extractions of the spent wood. Comparing results, the difference is due without doubt to the preparation of the wood; this point I have demonstrated very fully.

"The liquors obtained, especially the hemlock, were of much better color than from the Soxhlet.

"For the last four months, I have worked the percolator extractors alongside the Soxhlet, and under proper conditions, which are easily obtained, have no hesitancy in declaring my preference for the percolator system. Its simplicity in construction, and certainty of operation, together with its universal applicability, are important factors in its favor." .....

The ordinary Soxhlet extractor has been and is still popular as a means of extracting tanning materials, capable of boiling temperature extractions. The main objection being the continuous subjection of the constantly strengthened solution of extractives to the temperature of the flame under the reservoir. I believe that the higher non-tannins in the case of woods extracted by this method is due to the conversion of cellulose into glucose, and that the non-tannin figure would be as high in the extractions of the same material by Procter's method, were the initial and sustained temperatures of extraction the same in both cases. This condition exists in leaching on the commercial scale, and I have proven it to our own satisfaction on a scale between an analytical and a commercial basis.

In the case of barks the higher insolubles are undoubtedly due to the flame temperature, but so far I have not been able to demonstrate that the actual influence of the flame is as great as the supposed.

It has been suggested that the size of the cups and frequency of siphonings were factors in successful extraction by the Soxhlet method. The size of the cup has nothing to do with the frequency of siphonings. When extracting barks and light woods, the first siphoning is practically the only full cup siphoned. Subsequent siphonings are only fractional amounts of the capacity of the cup. The longer leg of the siphon is generally long enough to extend well into the reservoir, and on the first siphoning the siphon tube entrains alternate portions of air and liquor, and before the cup is nearly full the siphon will start and deliver whatever liquor has

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accumulated. A large cup Soxhlet when running on barks and light woods will siphon as frequently as an extractor having a small cup. Larger amount of liquor to a siphoning may be had by shortening the longer leg of the siphon so that it just engages the entrance to the reservoir.

The question of the thoroughness of a Soxhlet extraction will be touched on later in the report.

The removal of the first strong portions of extracted matter undoubtedly eliminates to some extent the possible reversion of tannin, by preventing the continued boiling of this stronger solution. Between 400 and 500 cc. seems to be the proper amount to remove in cases where a liter of water is used for extraction. The determinations I have thus far made by this method of extraction do not seem to show so great a difference in its favor as the supposed loss attributed to the old form of Soxhlet. Whether this is due to the imperfections of the improved method or to an exaggerated idea of the effect of the old method, I am not prepared to say. It is hardly safe to remove more than 500 cc. of the original liter because of the possible chance of leakage and consequent drying up of the reservoir.

The value of the Procter method for extracting Sumac, Algorabilla, Myrabolans, and kindred materials, is not to be questioned; but for use on oak and hemlock barks, chestnut, oak and quebracho woods, it appears that other methods involving less attention and trouble are better.

Mr. Kerr's percolation method requires a much finer preparation of the sample than is necessary for other methods, in order to effect a complete extraction in the minimum of time and with the minimum amount of water. I also found it difficult to regulate the supply of steam in order to insure a regular delivery of the condensation. To obviate this in using his method, I ran one end of a siphon tube down close to the bottom of the flask used as a steam generator for the extraction, ran the other end through a glass condenser jacket, and connected a water connection of the condenser jacket with the flask, so that steam surrounded the lower end of the siphon tube and kept the water at the boiling temperature. The small amount of drip from the condenser jacket was delivered to the extraction tube, and the main supply of

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extraction water was under perfect regulation by means of a screw clamp.

This method of extraction resembles in some respects Procter's method, differing, in that the solution passes directly through the column of tanning material, and the latter is not constantly surrounded by a solution of its own solubles. No heat other than that imparted by the extraction water is used and there is no jacket to conserve this heat so that the extraction is obviously at a point several degrees below the boiling point. With a well-regulated supply of hot water, and the advantage of finer preparation (which tends to hasten the extraction) it seems to give higher results than the ordinary Soxhlet, but the advantage over a Soxhlet extraction in which the first portions have been removed is not so marked. Compared with the latter method of Soxhlet extraction, I am not sure that it is more thorough. All extractions seem to me to be comparative rather than exact, and I doubt the ability of *any* extractor to remove *all* the tannin from, say, 25 gr. of bark in 10 hours, with a liter of water. At the expiration of that time the drip may seem clear, but subsequent leaching will not fail to remove small portions of soluble matter.

On the question of the thoroughness of the ordinary Soxhlet method, Mr. Kerr writes:

"With a view of determining to some extent the time necessary for a complete extraction of wood in the Soxhlet extractor, and also what proportion of the total extractive matter was removed in given periods, a portion of the official sample of spent wood was extracted and the resulting liquors drawn off, after 3, 6, 12 and 19 hours respectively. The results are herewith given:

	S S.	N. T.	Tan.
Portion 1, after 3 hours.....	2.75	1.05	1.70
" 2, " 6 "	1.36	.60	.76
" 3, " 12 "	1.37	.93	.44
" 4, " 19 "	1.12	.72	.40
Total results, 19 "	6.60	3.30	3.30

"Even after 19 hours active extraction, the removal of soluble matter containing tannin was not complete, as, when the extraction was continued some time longer, the resultant liquor still contained a very appreciable quantity of tannin.

"The difference between the above results and those obtained on the same wood, by 10 hours extraction, being so great, a check

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analysis was made which corroborated the results of the first 10-hour extraction, the average of the two being: S. S. 4.22, N. T. 1.79, Tannin 2.43. A comparison with those obtained after 19 hours shows an increase in the yield, not only of the soluble solids, but tannin, to the extent of over 56% and 35%, respectively. If this be true, (and some further trials which I also submit, seem to bear it out), a complete extraction of chestnut wood cannot be obtained in 10 hours, by the Soxhlet apparatus.

"It will be noted in the case of the 19-hour extraction, the purity has materially decreased; based on the soluble solids, the purity after 10 hours, is over 57%, whereas after 19 it is reduced to 50%. It is possible this decrease may be due to errors in the analyses of the liquors from the successive extractions, which were necessarily weak, but as they show an almost continuous decrease in the ratio of tannin to soluble solids, it is more likely that as the extraction progresses, the ratio of tannin becomes lower from some definite cause, which may be the solubility of the cellulose, due to the slight quantity of acid present.

"Table No. 5 gives the results above referred to, in which the extractions were made in two fractions of 9 hours each. So far as they go, they confirm the idea that 9 or 10 hours is insufficient for complete extraction in the Soxhlet apparatus. These analyses are from various samples of spent wood, as the official sample had been used up."

It is hardly fair to make the extraction of spent chestnut wood the basis of a test of the value of a form of extractor. The preparation of the sample exerts more influence on the extraction results in the case of wood, than in the case of bark. Again, while the extraction of *new* chestnut wood is, to say the least, puzzling, the extraction of *spent* chestnut wood which has been previously subjected to the influence of sustained high temperatures for probably 100 hours, shows this influence in the results, with the resulting paradox that attends the comparison of analyses of a new wood and the same wood after commercial leaching.

It appears, to me, better to use a bark as a basis of comparison. As I have said, I do not believe any extractor will thoroughly extract 25 gr. new bark with one liter of water in 10 hours; but I *do* think that the Soxhlet will come as close to doing it as any extractor.

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I extracted 25 grams of the new hemlock bark sent out, for 18 hours, changing the reservoir every 3 hours, and analyzed the different portions with the following results:

	S. S.	N.T.	T.
After 3 hours	16.248%	5.56%	10.688%
After 6 hours	1.572	.95	.622
After 9 hours	1.26	.78	.48
After 12 hours	.76	.66	.10
After 15 hours	.604	.59	.014
After 18 hours	.32	.32	.00
Totals for 18 hours	20.764	8.86	11.904

This was carried on in a glass Soxhlet with a glass flask for a reservoir, in order that observations of the progress might be made. The water in the reservoir was boiling constantly, and the successive flasks used were thoroughly cleaned.

With the copper Soxhlet the only way of knowing whether the water in the reservoir is boiling violently, is by the "rattle" in the condenser tube. If the heat is not sufficient at the start, or varies during the operation, the amount of condensation might become materially less and thus interfere with the operation. If from previously having boiled dry, the bottom of a copper reservoir is covered with a charred material, this material may become permeated with the soluble extractives, which may be removed by subsequent waters used to re-extract a residue, and because of the weak solution would exert an appreciable influence on the results.

I do not mean to insinuate that any of the above causes had any influence on Mr. Kerr's extraction, but am merely mentioning some experiences of my own.

A spent chestnut wood extraction conducted similarly to the bark extraction gave me the following results:

	S. S.	N. T.	T.
After 3 hours	1.77%	.87%	.90%
" 6 "	.48	.40	.08
" 9 "	.68	.53	.15
" 12 "	.28	.23	.05
" 15 "	.32	.30	.02
" 18 "	.388	.39	...
Total for 18 hours	3.918	2.72	1.20

A 14-hour Soxhlet extraction of the hemlock bark in glass, on two samples, one of which was just as sent out, and the other

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having been ground so that it would pass through an 8-mesh to the linear inch sieve, gave the following:

	S. S.	N. T.	T.
Regular preparation.....	18.92%	7.37%	11.55%
Fine "	19.18	7.63	11.55

Further extractions in the cases of the relay extractions of wood and bark would probably have resulted in more soluble matter, but I do not think that there would have been more than a trace of tannin.

Some of the difference between a straight 12-hour extraction and a relay extraction of the same period would probably be due to the continued boiling of the total extraction, which, of course, would not take place in the relay method.

As has been previously said, the work of this committee has been too scanty to warrant the adoption of any one form of extractor; but it appears that with a fine preparation, a limited height of column of material, a well-regulated supply of water, and the addition of a steam jacket, the Kerr apparatus must be considered as a meritorious method of extraction.

Between 25 and 30 grams of new oak or hemlock bark seems to be the proper amount for extraction with one liter of water. From 80 to 100 grams of these leached materials with one liter of water is probably the best amount for a good extraction, although with well-leached barks, this amount will not give a strong solution for analysis.

For quebracho wood I would suggest 20 grams and for chestnut wood 40 grams, as the proper amounts, and 80 grams for the spent woods.

Barks, woods, etc., for Procter and percolation methods should be ground as finely as possible. For Soxhlet extraction, a preparation that will pass through an 8-mesh to the linear inch sieve is recommended. It wil be impossible to prepare the fibrous portion of oak bark to conform to this condition, and I would suggest that oak bark receive the same treatment that reduces hemlock bark to this grade of fineness.

Dry material is, of course, more easily ground than damp, and when practicable, spent material should be dried at 100° C., then ground and the analysis conducted on a dry basis.

For new barks a moisture sample of 25 grams should be taken,

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and the analysis made with either absolutely dry or commercially dry material. I would suggest that new hemlock and oak barks analyses be reported on *both* "Dry" and "Commercially Dry" bases, and that 13% moisture be made the standard of commercially dry bark.

Samples of new bark should be taken at intervals as the bark enters the leach, placed in covered pails, mixed, and the laboratory sample taken from the mixture. Spent bark should be sampled at different levels from different parts of the leach, placed in covered pails, mixed and sampled; or, the spent samples may be taken at intervals from the conveyor carrying the material to the furnace room.

TABLE I.

New Hemlock Bark.

(a) Soxhlet Method.

Analyst	T. S.	S. S.	N. T.	T.	R	Quantity	Duration	Dry Hide
Eachus	21.10	18.78	7.68	11.10	2.32	25 gr.	10 hours	.....
Kerr.....	19.65	17.98	5.94	12.04	1.67	"	"	15.56 gr.
Teas.....	19.97	18.22	7.00	11.22	1.75	"	"	10. "
(b) Soxhlet, removing first portions of extracted matter.								
Eachus	20.36	18.40	6.63	11.77	1.96	25 gr.	10 hours	.....
Kerr.....	....	....	...	....	...	.....	.....	.....
Teas.....	20.90	18.50	6.52	11.98	2.40	25 gr.	10 hours	10. gr.
(c) Procter's Method.								
Eachus	18.90	17.35	6.15	11.20	1.55	.....	.....	.....
Kerr.....	....	....	...	....	...	.....	.....	.....
Teas.....	18.40	17.18	5.81	11.37	1.22	25 gr.	12 hours	10. gr.
(d) Kerr's Percolation Method.								
Eachus	20.38	18.40	6.89	11.51	1.98	25 gr.	10 hours	.....
Kerr.....	21.02	19.11	5.89	13.21	1.91	"	8 "	15.56 gr.
Teas.....	20.64	18.81	6.34	12.47	1.83	"	10 "	10.4 "
Averages.								
Soxhlet....	20.24	18.33	6.87	11.46	1.91			
" (Mod)	20.63	18.45	6.58	11.87	2.18			
Procter's...	18.65	17.26	5.98	11.28	1.39			
Kerr's.....	20.68	18.77	6.37	12.40	1.91			

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TABLE II.

New Chestnut Wood.

(a) Soxhlet Method.

Analyst	T. S.	S. S.	N. T.	T.	R.	Quantity	Duration	Dry Hide
Eachus	14.24	12.85	4.10	8.75	1.49	40 gr.	10 hours	.....
Kerr.....	15.21	13.81	3.64	10.17	1.40	"	"	15.94 gr.
Teas.....	14.56	13.23	4.24	8.99	1.33	"	"	10. "

(b) Soxhlet (Removing first portions of extracted matter).

Eachus	16.38	13.12	4.62	9.50	2.26	40 gr.	10 hours	.....
Teas.....	15.80	13.79	4.00	9.79	2.01	"	"	10. gr.

(c) Procter's Method.

Eachus	12.01	11.90	3.57	8.33	.11	40 gr.	10 hours	.....
Teas.....	11.50	10.60	3.02	7.58	.90	"	12 "	10.4 gr.

(d) Kerr's Percolation Method.

Eachus	16.21	14.21	5.10	9.11	2.00	40 gr.	10 hours	.....
Kerr.....	14.39	13.52	3.41	10.11	.87	"	8 "	15.94 gr.
Teas.....	14.60	13.34	4.04	9.30	1.26	"	10 "	10. "

Averages.

(a)	14.67	13.30	3.99	9.31	1.37
(b)	16.09	13.96	4.61	9.35	2.25
(c)	11.76	11.25	3.30	7.95	.51
(d)	15.07	13.69	4.18	9.51	1.38

TABLE III.

Spent Hemlock Bark.

(a) Soxhlet Method.

Analyst	T. S.	S S.	N. T.	T.	R.	Quantity.	Duration.	Dry Hide.
Eachus	4.36	3.41	1.77	1.64	.95	80 gr.	10 hours	.....
Kerr.....	3.96	3.48	1.68	1.80	.48	"	"	9.84 gr.
Teas.....	4.06	3.70	1.92	1.78	.36	"	"	10.

(b) Soxhlet (Removing first portions of extracted matter).

Eachus	4.97	4.57	2.49	2.08	.40	80 gr.	10 hours	.....
Teas.....	4.40	3.82	1.90	1.92	.58	"	"	10.

(c) Procter's Method.

Teas.....	3.60	3.21	1.48	1.73	.39	"	12 hours	10.

(d) Kerr's Percolation Method.

Eachus	4.99	4.50	2.60	1.90	.49	"	10 "	.....
Kerr.....	3.44	3.19	1.64	1.55	.25	"	8 "	9.84
Teas.....	3.90	3.38	1.70	1.68	.52	"	10 "	10.

Averages.

(a)	4.13	3.33	1.79	1.74	.60
(b)	4.68	4.20	2.20	2.00	.48
(c)	....	....	....	....	...
(d)	4.11	3.69	1.98	1.71	.42

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TABLE IV.

Spent Chestnut Wood.

(a) Soxhlet Method.

Analyst.	T. S.	S. S.	N. T.	T.	R.	Quantity.	Duration.	Dry Hide.
Eachus	4.48	3.88	1.80	2.08	.60	80 gr.	10 hours	.....
Kerr.....	4.29	4.03	1.66	2.37	.26	"	"	10. gr.
Teas.....	4.87	4.03	1.89	2.14	.84	"	"	10. "

(b) Soxhlet (Removing first portions of extracted matter).

Eachus	4.91	4.52	1.84	2.68	.39	80 gr.	10 hours	.. ..
Teas.....	5.21	4.33	1.78	2.56	.88	"	"	10. gr.

(c) Procter's Method.

Teas.....	3.10	2.84	1.10	1.74	.26	"	12 "	10. "

(d) Kerr's Percolation Method.

Eachus	4.90	4.61	2.30	2.31	.29	"	12 "	.....
Kerr (a)...	3.40	3.15	1.54	1.61	.25	"	8 "	10. gr.
Kerr (b)[1] ..	3.90	3.62	1.16	2.46	.28	"	8 "	10. "
Teas.....	4.20	3.84	1.74	2.10	.36	"	10 "	10. "

[1] Sample re-ground to pass through a 12 m. per inch sieve.

Averages.

(a).....	4.55	3.98	1.78	2.20	.57
(b).....	5.06	4.43	1.81	2.62	.63
(d).....	4.10	3.81	1.69	2.12	.29
(d) without Kerr (a)	4.33	4.02	1.73	2.29	.31

TABLE V.

	First 9 Hours.			Second 9 Hours.			Total (18 Hours).		
	S. S.	N. T.	T.	S. S.	N. T.	T.	S. S.	N. T.	T.
New Chestnut Wood..	12.71	3.84	8.87	1.53	.898	.632	14.24	4.738	9.50
Spent Chestnut Wood.									
No. 1.....	2.97	1.42	1.55	1.20	.483	.717	4.17	1.90	2.26
No. 2.....	2.80	1.37	1.43	1.20	.637	.563	4.00	2.00	1.99
No. 3.....	1.85	1.18	.67	.99	.71	.28	2.84	1.89	.95
No. 4.....	2.20	1.40	.80	1.06	.64	.42	3.26	2.04	1.22
No . 5..... :	4.62	2.15	2.47	1.23	.70	.53	5.85	2.85	3.00

WILLIAM H. TEAS,
Chairman.

The Referee not having the Committee Report from which to draw his conclusions, as stated, decided it would be best to request the various collaborators to extract two samples of materials, widely different in nature, and a sample of spent material, according to their laboratory method and any other method or

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methods they considered of value, trusting that so wide a scope for investigation would reveal the weak and strong points of several methods of extraction. This hope has been realized to a greater extent than was expected.

The instructions sent for general collaborative work are here given.

STUDY III.

Extraction of Tanning Materials.

The three samples furnished—Sumac, Quebracho wood and spent Oak bark—are to be analyzed according to the method employed by each individual analyst and by any other method or methods that seem worthy of trial. Rference is here made to the provisional method for the analysis of barks, woods, leaves, etc., made by last year's Referee, page 15 of the Report. In addition to the usual items of the analysis, which are to be reported upon a dry basis, the following data is requested:

1. Grams of material used.
2. Volume of liquor obtained.
3. Liquor, how diluted or concentrated.
4. Time of extraction.
5. Method of extraction.
6. Dry hide powder used per 200 cc.

The Referee would request that if any unusual method of extraction is employed, as full details as possible be given.

Attention is called to the fact that the Quebracho wood and spent Bark have not been prepared for analysis. Each analyst is requested to prepare according to his laboratory method, fully stating such method, with style of mill used for grinding. A 10-gram sample of the preparation is requested.

Twenty analysts collaborated on this Study and their results are tabulated below:

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TABLE XX.—STUDY III. FRESH QUEBRACHO WOOD.

Analyst	Total Solids	Soluble Solids	Insolubles	Non-Tannins	Tannins	Volume Obtained cc.	Volume for Analysis cc.	Dry Hide Gms	Method of Extraction	Amt. Ext'd Gms.	Time of Extraction, hours	Style of Grinder	Preparation, Meshes to Linear Inch	Notes
W. H. Teas,	28.60	26.38	2.22	2.90	23.48		1000	11.50	Glass Soxhlet	20	10	Enterprise Mill	8	Residue re-extracted gave .35 % Tannin.
H. T. Wilson,	32.60	29.22	3.38	3.04	26.18	900	1000	13.30	Siphon Soxhlet	20	16	"	20	
J. V. R. Evans,	26.71	24.69	2.02	2.97	21.72		1000	9.20	"	20	10	"	8	
W. K. Alsop and A. W. Hoppenstedt,	30.59	27.67	2.92	4.18	23.49	820	1000	11.40	"	15	10	"	8	N.T. shaken 4 min.
F. W. Ackermann,	30.26	27.75	2.51	3.97	23.78	870	1000	11.40	"	15	10	"	14	
Chas. Eachus,	28.99	28.68	.31	4.96	23.72		1000	9.78	Procter	15	8	Coffee Mill	14	
M. F. Nichols,	30.84	27.48	3.36	3.11	24.37	900	1000	10.20	Siphon Soxhlet	40	8	Enterprise Mill		
C. R. Delaney,	34.25	31.20	3.05	3.24	27.96	950	1000	11.80	"	10	10	"	20	
G. A. Kerr (1),	32.04	28.30	3.74	3.85	24.45		975	17.50	"	25	10	"	14	
G. A. Kerr (2),	32.53	28.00	4.53	2.51	25.49		1110	17.50	Kerr Percolator	25	10	"	14	
F. P. Veitch,	35.14	30.47	4.67	3.31	27.16		1000	11.80	Zulkowsky Continuous	15	20	"	25	1st portion ext'd at 60°—70° C.
H. H. Hurt,	35.27	32.03	3.24	3.59	28.44		1000	13.60	"	15	25	"	25	" " "
J. R. Mardick,		28.93		5.05	23.88	950	1000	8.60	Digestion & Decantation	25	6	Meat Chopper	12	
F. H. Small,	33.41	29.88	3.53	2.73	27.15	900	1000	12.70	Siphon Soxhlet	20	9	Enterprise Mill	14	
G. D. McLaughlin,	34.84	29.90	4.94	3.18	26.72	900	1000	13.00	"	20	16	"	20	
E. G. Wilmer,	29.36	26.47	2.89	2.49	23.98	950	1000	12.14	Digestion & Percolation	18	30	"	8	90°—100° C.
H. C. Reed, (1)	32.01	29.78	2.23	3.10	26.68		1000	13.00	Siphon Soxhlet	18	10	"	16	
H. C. Reed, (2)	34.13	30.58	3.55	3.08	27.50		1000	12.40	Reed Extractor	18	8.5	"	20	
H. C. Reed, (3)	35.50	31.46	4.04	3.29	28.17		1000	12.40	"	18	8.5	"	40	
H. C. Reed, (4)	36.12	31.91	4.21	3.61	28.30		1000	12.35	"	15	15.5	"	40	
C. W. Norris,	33.52	30.51	3.01	2.73	27.78	720		12.77	Siphon Soxhlet	27	8.5	Enterprise B. & Corn Mill	14	N.T. diluted 300 cc. to 500 cc.
F. R. Mosbaugh,		28.75		2.80	25.95		1000	13.20	Percolation	20	12	Disc Mill	20	Tailings concent'ed in vacuum.
W. A. Fox,	30.00	26.55	3.45	3.40	23.15	900	1000	10.00	Siphon Soxhlet	15	10	Enterprise Mill		
Average,	32.22	28.98	3.23	3.35	25.62		1004	12.24		19.5	12.2		18	
Maximum,	36.12	32.03	4.94	5.05	28.44		1110	17.50		40	30		8	
Minimum,	26.71	24.69	.31	2.49	21.72		975	8.60		10	6		40	
Greatest Diff,	9.41	7.34	4.63	2.56	6.72		135	8.90		30	24		32	

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TABLE XXI.—STUDY III. SPENT OAK BARK.

Analyst	Total Solids	Soluble Solids	Insolubles	Non-Tannins	Tannins	Volume Obtained cc.	Volume for Analysis cc.	Dry Hide Gms.	Method of Extraction.	Amount Extracted Gms.	Time of Extraction. Hours	Style of Grinder	Preparation. Meshes to Linear Inch.	Notes.
W. H. Teas (1),	8.67	7.41	1.26	3.93	3.48	...	1000	9.20	Siphon Soxhlet	50	10	Enterprise Mill	8	
W. H. Teas (2),	8.62	7.84	.78	3.97	3.87	...	1000	9.20	Modified Soxhlet	50	10	"	8	1st 450 cc. removed from extr.
H. T. Wilson,	9.89	8.70	1.19	4.39	4.31	700	1000	13.30	Siphon Soxhlet	100	16	"	14	
J. V. R. Evans,	8.67	7.41	1.26	3.93	3.48	...	1000	9.20	"	50	10	"	8	N. T. shaken 4 min.
W. K. Alsop and A. W. Hoppenstedt,	9.83	8.39	1.44	4.21	4.18	800	1000	11.40	"	60	10	"	8	
F. W. Ackermann,	9.26	8.04	1.22	4.07	3.97	950	1000	11.40	"	60	10	"	14	
Chas. Eachus,	8.56	7.00	1.56	3.62	3.38	800	1000	10.00	"	50	10	Coffee Mill	12	
H. F. Nichols,	8.69	7.67	1.02	3.89	3.78	900	1100	7.70	"	130	8	Enterprise Mill	....	
C. R. Delaney,	9.19	8.18	1.01	3.86	4.32	...	500	11.76	"	20	8	"	40	
G. A. Kerr (1),	8.43	7.12	1.31	3.64	3.48	...	995	17.50	"	60	10	"	14	
G. A. Kerr (2),	8.69	8.20	.49	3.56	4.64	...	1420	17.50	Kerr Percolator	60	12	"	14	
F. P. Veitch,	11.76	9.72	2.04	4.28	5.44	...	500	12.80	Zulkowsky Continuous	30	20	"	25	1st portion extracted at 60°—70°C.
H. H. Hurt,	11.72	10.01	1.71	4.55	5.46	...	500	13.60	"	30	25	"	25	"
J. R. Mardick,	.....	5.84	....	2.98	2.86	500	500	8.62	Digestion & Decantation	100	35	Meat Chopper	14	
T. J. Mosser,	8.65	6.75	1.90	3.61	3.14	600	1000	8.00	Siphon Soxhlet	60	8	.....	8	
G. D. McLaughlin,	10.68	8.89	1.79	4.34	4.55	620	1000	12.96	"	100	16	Enterprise Mill	14	
E. G. Wilmer,	7.78	7.60	.18	3.60	4.00	950	1000	12.14	Digestion & Percolation	50	30	"	8	90°—100°C.
F. H. Small,	10.70	8.11	2.59	3.42	4.69	450	500	12.70	Siphon Soxhlet	50	9	"	16	
H. C. Reed (1),	9.89	8.87	1.02	4.48	4.39	...	1000	7.50	"	50	10	"	16	
H. C. Reed (2),	10.55	9.85	.70	4.39	5.46	...	1000	8.90	Reed Extractor	50	15	"	40	1st 500 cc. removed from extr.
C. W. Norris,	9.49	8.24	1.25	3.77	4.47	820	....	13.43	Siphon Soxhlet	90	8.5	Enterprise Bone and Corn Mill	8	
F. R. Mosbaugh,	.....	8.72	...	4.53	4.19	...	1000	6.60	Percolation	50	12	Disc Mill	20	
W. A. Fox,	8.57	7.24	1.33	3.55	3.69	800	1000	10.20	Siphon Soxhlet	60	10	Enterprise Mill		
Average,	9.44	8.08	1.29	3.94	4.14	...	910	11.11		61.3	12.2		16	
Maximum,	11.76	10.01	2.59	4.55	5.46	...	1420	17.50		130	30.0		8	
Minimum,	7.78	6.75	.18	2.98	2.86	...	500	6.60		20	3.5		40	
Greatest Dif,	3.98	3.26	2.41	1.57	2.60	...	920	10.90		110	26.5		32	

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TABLE XXII.—STUDY III. SUMAC.

Analyst	Total Solids	Soluble Solids	Insolubles	Non-Tannins	Tannins	Volume Obtained cc.	Volume for analysis. cc.	Dry Hide Gms.	Method of Extraction	Amount Extracted. Gms.	Time of Extraction. Hours.	Notes.
W. H. Teas,	52.65	47.88	4.77	20.42	27.46	....	1500	11.50	Procter	15	13	600 cc. at 160° F., 900 cc. at 210° F.
J. V. R. Evans,	50.62	45.90	4.72	18.24	27.66	....	1500	11.50	"	10	14	
W. K. Alsop and A. W. Hoppenstedt,	50.87	47.88	2.99	18.40	29.48	1000	1000	11.20	"	10	3.5	N. T. shaken 5 min. Last 400 cc. at boil.
Chas. Eachus,	48.20	47.66	.54	19.22	28.44	....	1000	9.78	"	10	6	
M. F. Nichols,	50.23	45.91	4.32	24.26	21.65	1000	1100	8.10	Siphon Soxhlet	30	8	
C. R. Delaney (1),	49.63	49.63	.00	19.09	30.54	....	1000	11.25	Modified Koch Extractor	10	24	Laboratory temperarture to 100° C.
C. R. Delaney (2),	52.50	51.81	.69	20.08	31.73	950	1000	11.00	Siphon Soxhlet	10	8	1st five siphonings removed, then continuous
G. A. Kerr (1),	52.33	49.16	3.17	21.39	27.77	....	1000	14.00	" "	15	5	
G. A. Kerr (2),	54.30	50.45	3.85	19.60	30.85	....	1150	14.00	Kerr Percolator	15	5	
F. P. Veitch,	57.20	53.07	4.13	21.12	31.95	....	1000	11.80	Zulkowsky Continuous	12	20	1st portion 60°—70° C.
H. H. Hurt,	57.02	52.97	4.05	19.72	33.25	....	1000	13.60	" "	12	25	" " " "
J. R. Mardick,	.....	43.33	...	16.85	26.48	950	1000	8.60	Digestion and Decantation	25	6	
T. J. Mosser,	.....	45.84	...	18.67	27.17	....	2000	8.00	Percolation	25	15	90° C.
E. G. Wilmer,	46.65	44.82	1.83	20.40	24.42	800	1000	12.41	Digestion and Percolation	15	20	90°—100° C.
F. H. Small,	46.99	46.09	.90	17.60	28.49	1500	1000	12.70	Percolation	16	..	50°—55° C.
H. C. Reed (1),	48.55	47.92	.63	18.67	29.25	....	1000	12.90	Reed Extractor	15	10	By percolation up to 80° C.
H. C. Reed (2),	59.27	56.27	3.00	24.55	31.72	....	1500	9.50	" "	13	15	500 cc. outside.
H. T. Wilson,	57.03	54.27	2.76	24.14	30.13	1200	1055	12.92	Digestion and Filtration	15	15	90°—92° C.
F. R. Mosbaugh,	.....	40.07	...	18.91	21.16	....	1000	13.20	Procter	15	12	Tailings concentrated in vacuum. Tempt. extraction 70°—80° C.
W. A. Fox,	50.00	46.92	3.08	18.30	28.62	1000	1000	10.80	"	10	4	
Average,	52.00	48.39	2.67	19.98	28.41	....	1140	11.44		14.9	11.3	
Maximum,	59.27	56.27	4.77	24.55	33.25	....	2000	14.00		30	25	
Minimum,	46.55	40.07	.00	16.85	21.16	....	1000	8.00		10	3.5	
Greatest Difference,	12.72	16.20	4.77	7.70	12.09	....	1000	6.00		20	21.5	

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STUDY III.

Collaborators' Notes and Suggestions.

W. H. Teas.

"Fifty grams of the Spent Oak were used in order to make duplicate analyses. It is our usual practice to extract 80 grams, although this amount does not bring the tannin within the prescribed limits.

The Quebracho charge of 20 grams is our usual amount for this material. This was extracted in glass, and although the drip seemed clear to the eye, a further 10-hour extraction of the residue gave some tannin."

W. K. Alsop and A. W. Hoppenstedt.

"*Spent Oak Bark.*—Solutions were shaken, let stand ½ hour, 100 cc. taken for total solids; 150 cc., with 2 grams kaolin, thrown on 18 cm. filter and all allowed to filter through; fresh solution thrown on filter and 100 cc. of filtrate evaporated for soluble solids. Forty grams of wet hide were shaken 4 minutes, with 200 cc. of solution, thrown on 1 filter after shaking up with 2 grams kaolin; 100 cc. clear filtrate evaporated for non-tannins.

Quebracho Wood.—The details of the analysis of Quebracho wood are the same as under Spent Oak Bark. We are of the opinion that some better method of extraction should be used than the siphon extractor. It is admittedly unsuitable for some materials and has other faults besides. The rate at which the water is boiled has a very marked effect upon the results obtained, and it is very difficult to regulate the flame so as to obtain uniform results, as it is impossible to see what is going on inside of the extractor."

Sumac.—Mr. Alsop states that with 8.4 grams dry hide the non-tannins were 19.12%, and with 11.2 grams dry hide, 18.40%, and with 14 grams dry hide, 18.34%, the three amounts of dry hide representing 30, 40 and 50 grams wet hide, respectively. He says, "Our usual method is to use 40 grams."

"You will notice that there is practically no difference in N. T. when 40 and 50 grams of hide were used, and that 30 grams gives higher N. T. Counting the N. T. obtained with 40 grams hide as correct, the solution contained .2712 grams, Tannin per 100 cc. Taking .400 gram per 100 cc. as the strength to be used for

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12.5 grams dry hide, this solution should have required 8.46 grams hide. The 30 grams wet hide used contained 8.40 grams dry hide. Some time ago we analyzed a sample of Sumac, which contained .2320 gram, Tannin per 100 cc., equal to 7.25 grams dry hide to 200 cc. 30 grams wet hide containing 7.65 grams dry, gave N. T. 23.13%; 40 grams wet hide containing 10.20 grams, dry, gave N. T. 21.30. Of course, you will understand that these samples were analyzed by the laboratory method used here, and that no attempt was made to make the solution Official strength, nor were they shaken 10 minutes."

H. T. Wilson.

"*Sumac.*—Fifteen grams were digested on steam-bath with six successive 200 cc. portions of water, 2 to 3 hours being given for each digestion. After each digestion the whole mass was thrown into a perforated funnel and sucked dry with a vacuum pump."

C. R. Delaney.

Mr. Delaney used a modified Koch extractor. He says: "The method has its advantages, but for Sumac extraction I prefer the Soxhlet used properly by removing the first five siphonings before they have boiled and only allowing the latter ones to boil for any extended time. I have made a number of tests with the percolator against the Soxhlet on identical samples, and in only one case has the percolator come up to the Soxhlet extraction either in extract or tannin."

G. A. Kerr.

"I have endeavored to ascertain the time necessary for an absolute extraction of tannin from wood, but have, so far, failed to do so. In one case, the extraction was continued for 45 hours, consecutively, the liquor being removed every 9 hours, and fresh water applied, at the end of this time, there still remained appreciable quantities of tannin in the liquors. In fact, there seemed to be little diminution in the yeild, during the last 18 hours.

"I am of the opinion, now, that preparation of the material is an important function, and the completeness of extraction in any form of extractor, within reasonable time limits, depends upon the fineness of the reduction. I should not be surprised, if, grinding all materials as fine as sumac, and mixing with sand,

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powdered glass or quartz, would result in more complete extractions in the same length of time now employed.

Last year, during discussion of preparation, the point was made, that chestnut oak bark could not be ground to pass a fine meshed sieve. I find there is no difficulty whatever in grinding dry oak bark in an ordinary Enterprise drug mill, so fine that it will pass a sieve 20 meshes per inch. From such figures as I have been able to make, the indications seem to be that some decomposition of tannin takes place in the Soxhlet extractor, as my tannin results are, with one or two exceptions, higher in the percolator, than the former. This is also true of completeness of extraction. While the volume of liquor is somewhat higher, the results are obtained in the same length of time, or less. This is notably so with sumac, the liquor from which suffers considerable deterioration from boiling over a direct flame. Personally, I do not consider the Soxhlet extractor, as applied to tanning materials, adapted for the extraction of sumac.

"I do not think comparative results are to be looked for on the spent chestnut oak bark, unless the Referee took the pains to apportion the same proportion of lumps to each collaborator. The sample we received consisted of lumps, stringy inner bark and dust, making it a hard matter to sample accurately."

Mr. Kerr's percolator is fully described in last year's report.

F. P. Veitch.

"In extracting materials we always obtain a portion of the percolate at about 60° to 70°. The extractor is then connected with the condenser, and the extraction continued at steam-heat, the contents of the receiving flasks being removed as the liquor becomes concentrated and replaced by water. This operation is repeated until extraction is complete, which we find to require from 16 to 25 hours. As a matter of fact in every case we found that by again extracting for several hours with water we got some tannin. The reaction was not marked, however."

J. R. Mardick.

"We grind the bark or wood just as we receive it in a common meat crusher or chopper. This simple instrument prepares the bark, or spent bark (50% moisture) in a most convenient way. For the extraction of tanning material we simply cover the

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material with water in a beaker, digest at 100° C., for half an hour, then decant through 100 mesh brass sieve, return the bark again to the beaker, cover with water and continue digestion for ½ hour. For fresh bark, after decanting 5 times, we squeeze the spent bark in cheese cloth 5 times, till a litre solution is obtained. In this way we get a clear tannin solution without letting cloudy or colloidal filtrates through. The results compare very well with ordinary copper Soxhlet, with many advantages in its favor." favor."

E. G. Wilmer.

"Between 18 and 19 grams of the Quebracho wood and 50 grams of the Spent Oak Bark are placed in copper extractor and covered with 500 cc. water at 90° to 100° C. After standing 24 hours, contents of extractor are again heated by means of steam and then allowed to percolate through a plug of cotton into bottle. Percolate to 950 cc., adding hot water as needed. Liquor is now allowed to stand over night and made up to 1,000 cc.

Sumac.—Fifteen grams placed in quart bottle and 800 cc. water at 90° to 100° C., poured into same. Bottle and contents placed on steam-bath and with frequent shaking, allowed to extract for 20 hours, cooled and contents filtered through plug of cotton and made up to 1,000 cc."

F. H. Small.

"*Sumac.*—The sumac was intimately mixed with an equal volume of sand, placed in a water jacketed percolator and extracted at 50° to 55° C. 1,500 cc. were run through, the first 900 cc being saved apart and the last 600 cc. concentrated to 100 cc., and added to the 900 cc."

Chas. Eachus.

"In the analysis of the sumac, I extract in an extractor such as Procter describes. It consists of an inverted thistle tube with cloth over the bulb and is covered with sand in a copper beaker which sets in water heated to the required temperature. I find this apparatus extracts sumac uniformly and I get very close soluble solids on duplicates. It took 6 hours to extract the sumac but I can extract as well in 2 hours. The Quebracho wood was extracted exactly the same way, with the same quantity of material and water. It took from 6 to 8 hours to extract it."

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C. W. Norris.

"For grinding samples 'The Enterprise Bone and Corn Mill No. 750, was used. This mill consists of a fixed and a rotating disc and having alternating rings of teeth.

"The Spent Oak Bark was first sifted and the course part was run through the mill; then the long fibers remaining were cut with a pair of shears. In this way I was able to obtain a fairly uniform sample."

Irrespective of the style of extractor, the manner in which it is used, and the quantity extracted, there are two elements that determine the excellence of inferiority of the extraction,—preparation and duration of extraction. The coarser the preparation the longer the time required for complete extraction, and *vice versa*. But as the time for extraction has a distinct bearing upon the character of the extractive, continued heating resulting in destruction of tannin, the logical conclusion would be to shorten the time of extraction by increasing the degree of fineness of the preparation.

Consultation of the tables showing the results on the fresh Quebracho wood and spent Bark will disclose that the percentage of extractive and the tannin content increases in direct proportion to the fineness of the preparation, even taking into consideration the variety of methods of extraction used. The Referee has compiled a table from the general tables on the Fresh Quebracho Wood and Spent Bark to bear out this statement.

Table Showing Average Increase of Extractive and Tannin with Increase in Degree of Fineness of Preparation.

TABLE XXIII.

		8 Mesh	12 Mesh	14 Mesh	16 Mesh	20 Mesh	25 Mesh	40 Mesh
Quebracho Wood	T. S..	28.81	....	31.79	32.01[1]	33.96	35.20	35.81
	T. ..	23.17	23.88[1]	25.40	26.68[1]	26.86	27.80	28.24
Spent Bark	T. S..	8.82	8.56[1]	9.39	10.30	8.72[1]	11.74	10.55[1]
	T. ..	3.80	3.38[1]	4.19	4.54	4.19[1]	5.45	5.45[1]

[1] Indicates single analysis.

It does not necessarily follow that all tanning materials should be ground to an equal degree of fineness in order to insure a maximum extraction, as the character of the material has much to do with the ease or difficulty of extraction. Thus, in the case of the fresh Quebracho wood a 40-mesh preparation is apparent-

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ly none too fine, while with the Spent Bark a 25-mesh preparation is probably sufficiently so.

The Referee contends that much of the variation in analyses of barks, woods, etc., is directly traceable to incompleteness of extraction, and that it is only by approximating complete extraction that fairly comparative results can be looked for. With this in mind, and considering also the impractibility of defining the degree of preparation for each tanning material, he would recommend that tanning materials should be ground for extraction, to pass a screen of 20 meshes to the inch. It would seem inadvisable to stipulate a fixed time for extraction, more than to advise that it be sufficiently extended to approximate complete extraction. Sufficient data is given in the report of the "Extractive Committee" to prove the evil effects of prolonged boiling upon tannin solutions, and therefore it would seem advisable to abandon the copper Soxhlet form of extractor, so extensively used, and adopt some modified form which will admit of the removal of the first portions of the extractive solution.

If it is agreed to use a preparation as fine as has been recommended, this first portion will contain an exceedingly high percentage of the total extractable matter and but little less of tannin will result from the continued boiling of the more difficult extractable matter which remains.

The form of extractor to be used cannot conscientiously be more specifically defined by the Referee, as he has designed an extractor himself and does not wish to appear as though making the use of it in any way obligatory. The extractor will be considered in a separate paper.

It would seem best not to alter the method which calls for the extraction of such a quantity as will give from .35-.45 grams tannin per 100 cc. with fresh materials. However, it is the Referee's experience that when complete extraction is approximated no important difference will result when smaller quantities are extracted than provided by the official method if the amount of hide powder for non-tannin determination is reduced proportionally. The report of the Extractive Committee favors the extraction of 80 grams for spent tanning materials, but the Referee contends that the results of the extraction of the Spent Oak Bark prove the amount taken is but a secondary consideration, and believes, as he stated above, that if the quantity of hide powder is rightly ad-

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justed, a variation, within reasonable limits, in amount extracted can be safely ignored.

Incidentally the Referee would call attention to the fact that he has found the time contact method for soluble solids filtration recommended by Mr. Small of much value in the analysis of crude tanning materials, in that it enables one to obtain duplicate soluble solids and non-tannins, with a single total solids, from a liter solution.

The work that has been done this year on Sumac extraction seems to be in some measure contradictory to our previous understanding of that subject. The assumption that the temperature of extraction should not rise above 80° C., in order to obtain the maximum percentage of tannin, as noted in the Referee's report of last year, is not borne out by collaborative work. Prof. Procter, in his "Principles of Leather Manufacture," states that the maximum percentage of tannin is obtained from sumac when the temperature of extraction is between 50° and 60° C., but such a temperature limit seems to be utterly disproved by the results of Messrs. Delaney, Veitch, Alsop and Hurt, and the results of the Referee as well. On the other hand, the continued boiling of that portion of the solution containing the readily extractable matters of Sumac seems to work greater harm to the tannin than the continued boiling of the majority of tannin solutions, as is shown by the extraction of Sumac in the continuous copper Soxhlet extractor.

So with Sumac, as with other tanning materials, the removal of the first fractions of extractive solution with subsequent continuous extraction seems productive of the best results. In the analysis of the Sumac extreme care should be observed in correctly apportioning the hide powder to the tannin content, as a small variation in the quantity of hide powder seems to produce wider variations in non-tannin figures than with other tannin solutions.

With respect to the mill used for grinding tanning materials, it will be noted that the majority of the collarborators seem to prefer the Enterprise. The Referee is fully cognizant of the fact that the labor of preparing the material so that it may pass a 20-mesh screen, is considerable, inasmuch as the whole amount of a given sample must be ground to pass the screen and no portion dis-

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carded. He is, however, convinced that even with stringy materials this result can be effected with care and patience.

A mill devised on the principle of cutting instead of grinding, would aid us materially.

STUDY IV.

Under this Study, the Referee sent out the following instruction:

The Referee desires that color tests be made of the sample of Hemlock extract, according to the method employed by the individual analyst.

Collaborators' Notes and Method of Making Color Tests.

G. A. Kerr.

"The enclosed skiver was tanned in accordance with our usual practice, when we have only a limited quantity of extract with which to work. When testing our own extract, quick results are frequently desirable, and we proceed in a slightiy different manner, which is described below.

"Skiver submitted was first soaked for an hour in clear tap water, then thoroughly worked by hand, till well broken up and softened, being washed several times in fresh water during the working. Excess water was then squeezed out by hand and the skin allowed to soak in a dilute solution of borax for 30 minutes, after which it was washed in two clear waters.

"Tanning was done by shaking gently on shaker for 30 minutes, with a liquor of 5° Bark. which was then strengthened to 10°, and shaken 30 minutes more. By this time, color on both sides is fairly set, and we then made up a 20° Bark., liquor with lukewarm water, allowed it to cool to near laboratory temperature, and immersed the partially tanned skiver over night. The tanning completed, we rinse off in warm water, tack on board, and dry in a cool, dark place.

"When skiver test is desired quickly, we prepare skin as above, but shake in 5° and 10° liquor only 15 minutes. We then immerse skin in 80° liquor and knead the skin, by hand, for 30 to 45 minutes, when it is thoroughly tanned. The method of drying is the same, only previous to tacking on board, we roll the tanned

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skiver in a folded towel, and put under press for a minute or two; in this way, drying is cut down to a few hours. We frequently turn out tanned skivers in less than *8 hours.*

In addition to the hemlock skiver, we send you two skivers, made from the official sample of sumac. You will note a great contrast in color, which we find is a characteristic difference between liquors made in the Soxhlet and percolator extractors. It occurs to a similar extent in the extraction of all tanning materials.

"Referring again to the matter of color tests, we send you for comparison four 'swatches,' familiar to those who handle dye stuffs, which were dyed with four different brands of oak extract. You will note the delicacy in gradation of shade in each of the four, as shown in the center strips. Repeated tests on these same extracts result in identical results. Whether or not this test can be applied to all tanning materials, I do not know, but as oak extract is the principal product in that line and the one on which color is of real importance, we believe this suggestion may prove worth looking into.

"It might be possible to establish a basis of color value by this means, which can never be attained by the use of sheep-skin."

E. G. Wilmer.

"A small piece of bated skin is suspended for 48 hours in solution of the extract, containing about .6% tannin. The partially tanned sample is then suspended in a 1.5% solution and when well tanned, is washed, treated with sperm oil and allowed to dry."

W. H. Teas.

"Three samples of color tests made from the hemlock extract are sent; 'A' was shaken 20 minutes in part of the solution used for analysis, then placed in a fresh portion of the same solution over night. In the morning it is washed, wrung out, stretched on a board and dried in a cool, dark place

"'B' had the same treatment, except that a 20° Bark. liquor made from the extract, took the place of the weak liquor used for 'A.'

"'C' was oiled while wet, with a mineral leather oil."

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M. F. Nichols.

"Color test Hemlock extract. Tan pickled sheepskin by starting with about ½% tan solution with addition of little salt. After 12 hours strengthen solution to 1% and tan over night, wash and dry in dark place."

H. T. Wilson.

"Raw skin is soaked in fresh water over night and well cleaned. A ½% solution shaken from 10 to 15 minutes and again shaken in the same strength solution for the same time, transferred to a 1% solution, shaken 30 minutes, then placed in a 2% solution, and at night transferred to a 4% solution and allowed to remain till tanned. It is then tacked up and dried.

F. P. Veitch and H. H. Hurt.

"For the color determinations Lovibond tintometer is used."

The Referee uses sheepskin for his color tests, washing first thoroughly with water and then with a very dilute solution of borax, allowing to soak in this solution over night. In the morning the skivers are well rinsed with fresh water and thoroughly worked in water containing a trace of acetic acid. After thorough rinsing, the skivers are shaken for 5 minutes in a tannin solution of the strength used for analysis, and again shaken for 10 minutes in a 2% solution and the color allowed to set in this solution. Skivers are then laid away over night in a 25° Bark. liquor, thoroughly rinsed the next morning, tacked on a board and allowed to dry in a dark place.

The Referee has mounted the color tests made on the Hemlock extract, by the various analysts, and submits them for your inspection. That there would be a considerable variation in results, was to be expected where there are so many variations in method.

Different portions of the same skin will produce different shades with the same extract, and it is not surprising that different skins, tanned by different methods, fail to produce uniformity. It is probable that a fixed method for color tests where skin is used, can never more than approximate uniformity. It may be that some such scheme as the one proposed by Mr. Kerr, where the color is precipitated as a lake by the use of a mordant, will solve the problem.

It is recommended that the entire matter be made a subject of

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investigation next year, and that in addition to the lines of work indicated, the usefulness of the tintometer for the purpose be investigated.

The work this year has plainly evinced the need of such investigation.

GENERAL.

No report was received from the Committee on Acid Determination, and the Referee very much regrets that this important subject yet remains in so unsatisfactory a condition.

The charcoal method gives, with some liquors, probably approximately correct results, but with others this is far from being the case. Moreover, phenolphthaleïn serves as a poor indicator for certain liquors. The spot test on litmus method presents the advantage of permitting the employment of stronger solutions for the analysis, thereby lessening the chance of error. The Referee has thought that in the place of litmus paper, passibly a test paper made by soaking with a phenolphthaleïn solution, might answer better, proving more sensitive, but has had no opportunity to make experiments.

Of the Committee appointed to investigate the Parker-Payne Method of Tannin Analysis, Mr. Wilson was the only member to respond. The report is here inserted.

COMMITTEE REPORT ON PARKER-PAYNE METHOD.

Two samples of extract were sent to the members of the committee with instructions to analyze according to the Parker-Payne method the pure extracts, and to make further analyses by this method after the addition of small known quantities of acetic and lactic acids.

The chairman failed to carry out his own instructions and has no complaint to make because only one member of the committee responded. My views on this method were covered by the paper read at the last meeting of the Association, and I am still of the opinion that the authors were a little hasty in publishing the method in its present form.

Mr. Wilson was the only faithful member of the Committee and I take pleasure in presenting his results.

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	% Tannin by H. P. method.	% Tannin by Parker-Payne. Total absorption.	Total absorption in cc. $Ca(OH)_2$ for 100 cc. liquor.	Acid absorption cc. $Ca(OH)_2$ for 100 cc. liquor.	True Tannin cc. $Ca(OH)_2$ for 100 cc. liquor.	% True Tannin.
Chestnut Extract	31.30	a31.01	54.25	....	25.85	14.77
	....	.....	....	28.4	....	....
	....	b31.29	54.75	....	26.35	15.06
Chestnut Extract plus ½ % acetic	....	a27.70	48.60	....	20.45	11.65
	....	.....	....	28.15	....	....
	....	b27.98	49.10	....	20.95	11.93
Quebracho Extract	38.26	a28.10	35.25	....	23.65	....
	....	.....	....	11.60	....	....
	....	b28.10	35.25	....	23.65	18.84
Quebracho Extract plus ½ % acetic	....	a25.09	31.85	....	19.50	15.36
	....	.....	....	12.33	....	....
	....	b26.08	33.10	....	20.75	16.35
Tannery Liquors from leaches	6.43	5.59	36.6	18.	18.6	2.83
Tannery Liquors:						
3″ New	4.51	4.18	54.0	26.8	27.2	2.10
3″ Sap	4.20	4.10	53.	25.2	27.8	2.15
Head Rocker	2.34	2.55	49.	36.4	12.6	.65
Tail "	1.19	1.44	55.	42.8	12.2	.31
New Bark	9.22	9.08	36.	22.8	13.2	3.32
Spent Bark	2.38	2.40	32.	25.2	6.8	.51

WM. H. TEAS,
Chairman.

It is to be regretted that so little investigation has been carried on this year on the Parker-Payne Method. Since Mr. Wilson's results stand alone, uncorroborated by returns from any other members of the Committee, the entire subject must be laid over to another year, and the Referee so recommends.

The Committee on the Revision of the Official Method of Analysis, of which the Referee was Chairman, thought it best to defer the revision, since the findings from this year's investigations might have no inconsiderable influence in altering the method. Therefore, in place of a committee report, the Referee has appended to this report the Official Method, so revised as to permit the ready framing of a complete and simplified Official Method, if it be considered advisable.

RESUME OF RECOMMENDATIONS EMBODIED IN REPORT.

It is recommended that the following questions be made subjects of collaborative research during the ensuing year:

1. Soluble Solids filtration, with especial reference to the time

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contact method, so termed, in conjunction with the temperature problem.

2. Analysis of liquors and the effect of the acidity of liquors upon the analysis.

3. Estimation of acid in tan liquors with a view of improving the present method.

4. The influence of acidity and alkalinity upon the chroming of hide powder and upon the subsequent analysis. The elimination of the sulphate factor and the merits of a fully or partially chromed prepared hide powder.

5. Extraction, for the purpose of confirming or rejecting the recommendations of the Referee.

6. The Parker-Payne Method of Tanning Analysis.

7. Estimation of nitrogen in leather and tan liquors.

It is further recommended that the following methods and processes be adopted provisionally:

8. The chroming of hide powder by the addition of the entire amount of chrome alum at one time.

9. Limiting the moisture content of wet chromed hide powder used for analysis between 70 and 75%.

10. The analysis of liquors as recommended by the Referee.

11. The form of apparatus known as the combined evaporator and dryer.

And it is finally recommended that,

12. An Official Method be drawn up from the present method, including the Provisional Methods, recommended in this year's report.

In conclusion the Referee desires to express his keen appreciation and sincere thanks to all who have so generously and unselfishly contributed to this report, and especially to the chairman of the several committees, who have labored so earnestly during the past year for the advancement of our knowledge, and the betterment of the processes under which we are working.

H. C. REED,
Referee.

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THE WASHINGTON CONVENTION.

The Convention of the American Leather Chemists' Association at Washington was called to order by President W. H. Teas at 10.30 A. M., November 15, 1905, at the Normandie Hotel.

The following were present:

T. J. Mosser, Newberry, Pa.

J. H. Russell, Philadelphia, Pa.

W. K. Alsop, New York.

F. H. Small, Worcester, Mass.

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Dr. V. C. Casaburi, Wilmington, Del.
R. P. Cushing, Lock Haven, Pa.
Wm. H. Teas, Ridgeway, Pa.
C. W. Norris, Milwaukee, Wis.
H. T. Wilson, Beuna Vista, Md.
F. P. Veitch, Washington, D. C.
H. H. Hurt, Washington, D. C.
C. C. Smoot, Alexandria, Va.
H. C. Reed, Stanford, Conn.
Oma Carr, Canton, N. C.
Alan A. Claflin, Boston, Mass.
E. J. Haley, New York.
Jno. H. Yocum, Newark, N. J.
C. C. Smoot, III, Alexandria, Va.
C. N. Proctor, Boston, Mass.

PRESIDENT'S ADDRESS.

Gentlemen of the Association:

It is not my purpose in opening this convention according to program with an address, to aid in forming a precedent as to the character of the address. I feel that we shall have enough papers of an academic nature, and that your president should confine himself to a short talk on conditions and progress, and the recommendation of features, which from his point of view, would help the growth and importance of the Association.

The conclusion of the second year of our association finds it growing and in a most prosperous condition; a child already on its feet; and although it is not quite two years old, it is even now insisting on running alone. But, gentlemen, while we rejoice at the energy of this forward child, let us watch it carefully, so that the ardor of its precocious youth does not sap the vital strength for adult life.

A year ago we had 19 active and 9 associate members; today there are 24 active and 18 associate members. This growth in membership is indeed gratifying to note, and the growth of enthusiasm in the objective work of the association is a further indication of the success and permanency of our organization. So

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long as the present interest is maintained, there need be no fear regarding the future life and influence of the association, and all that is needed to maintain the interest is the honest endeavor of every member to advance the efficiency of the leather chemist's laboratory as regards its analytical results.

The object of the A. L. C. A. is solely the advancement and standardization of methods of analysis; we are not organized for the purpose of solving manufacturing problems, nor to inquire into methods of manufacture. Our announced field is broad enough to preclude the possibility of the laborers, as an organization, getting in the adjoining field of manufacture. This being true, there should be no hesitancy on the part of those interested in joining in the work and assisting in the realization of the betterment and standardization for which we are working.

Until our association was formed, America was without an organization whose sole object was that of this association, and the fruits of the labors of a comparatively few leather chemists were credited to a larger body doing commendable work along other lines. Our kindred Continental organization enjoys the confidence and respect which comes with age and honest labors, and our own association can easily attain a similar or even higher position on this side of the Atlantic, by honest intelligent work. Publicity will materially aid us in this work, and a regular publication containing results of the investigations of the association, and of individual members, will undoubtedly develop helpful criticism, provide the members with a forum, and the association with a representative and official organ. The work necessitated by such a publication would not be so great as is generally imagined; it could be issued as a quarterly and contain besides our own efforts, the contemporary work of foreign chemists in our line, and would thus become an epitome of the various published papers relevant to our subject.

A board of three editors could divide the work, and if two were selected with regard to their respective proficiency in French and German, the third could act as compiler of the association work and of other articles in our own language. The report of our annual meeting would in itself make a very respectable number, and the other three numbers would then cover the entire year.

The scheme I have just outlined was developed in conversation

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with some of the members, and I trust that the association will give it serious consideration, and decide that a publication is essential to the progress of the organization.

Just a word regarding our by-laws: They are not sufficiently clear on some points, and not as complete as they should be, and I hope that this convention will interpret doubtful clauses and make the code more complete.

The results of the collaborative work of this year will, of course, be reported by the Referee, and I need only remark that in volume at least, it surpasses any previous year's collaboration. If the association decides to publish a quarterly, it would in my opinion, be a wise move to apportion the collaborative work, so that a different topic could be taken up three or four times during the year, and thus do away with the present system of practically all topics at the same time. The proposed arrangement would distribute the work throughout the year, and the published results would be before us while the work was comparatively fresh in our minds, and deductions would be correspondingly impressed. This system of distributed work would not be so apt to become a burden on the busy members of the association, as is often the case when an annual collaboration requires three or four days consecutive work. However willing to participate, it is often the case that a member's routine duties are such that protracted interruption is not welcome and often inconvenient. This suggested arrangement would probably increase the total volume of the work of the Referee, but the benefits of distribution and specialization would hold good in his case as well as in the case of the individual workers under his direction.

Apropos of our analytical work, I want to urge on the members the importance of close observance of adopted methods of analysis. Although it has been discovered that the accepted official method of the A. O. A. C. has, through oversight, never been made official, we regard it as official because individual and collaborative experiments have indicated its provisions as the best known method of tannin analysis, and this is all that can be hoped for in any future official method. The comparative results of American chemists on a given sample of tanning material will stand a favorable comparison with work done by European chemists under similar conditions. I believe that there will be less dif-

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ference in the analyses of a sample made by two American chemists, than in the analyses of the same sample by two European chemists under similar conditions.

The difference in results, whatever it may be, is generally due to a lax observance of the details of the method; and if we are to attain the highest concordance in collaborative and commercial work, the letter of the method must be fulfilled. Temperatures of filtrations, methods of drying, treatment of hide powder, etc., should be exactly as provided. If the individual idea on a point in the method differs from the prescribed, collect data and evidence in proof of its superiority and bring the matter before the association, but meanwhile, follow the official method on every analysis, the results of which are to be known as having been obtained by the official method. Local and personal improvements on the methods should not enter into "official" analyses, as, obviously these will not improve concordance. Improvements of proven universal merit will always be incorporated in the official method, and the judgment of the entire body is generally safer than that of the individual.

I cannot close without a brief expression of sincere regret that death has deprived us of one of our most valuable members and ardent workers, George P. Craighill, in point of age, was the father of us all, and yet in spirit he was as youthful and understandingly sympathetic as the youngest. A gentleman of the old school by instinct and breeding, it was indeed an honor to have the friendship of such a man, and a privilege to have been associated with him in our common endeavors. But he has left better titles to remembrance than any which we can bestow by eulogy. There was an unsullied purity in his private life; there was an inflexible integrity in his business conduct; an indescribable fascination in his familiar conversation; a quickness of perception and deduction; a direct and vigorous purpose in all that he wrote or did, and withal a superb dignity of deportment which together made a character that cannot fail to be contemplated and admired by those who knew him as long as we shall admire such qualities.

The association has indeed lost a valuable worker and the members a true friend, and I am positive that the sentiments of my humble contribution to the memory of the Grand Old Man of our organization are shared by all.

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At the conclusion of the President's address, the minutes of the last meeting were read and adopted.

The Secretary's report was read and filed.

The Treasurer's report was filed as read, and is as follows:

REPORT OF THE TREASURER FOR 1904-1905.

Balance on hand September 26, 1904	$ 40.35	
RECEIPTS		
By subscription for stenographic report, 1904	$ 50.00	
By annual dues for 1905	185.00	
By annual dues for 1906	10.00	
Total receipts for year 1904-'05	$285.35	$285.35
EXPENDITURES		
To Leonard & Satterlee, stenographic report	$ 85.00	
To Hide & Leather, 250 copies of report	60.00	
To J. Owen Harvey, flowers	10.00	
To printing, postage, stationery and telegrams	20.39	
To Portway & Co., filter candles	12.86	
To expressage for Referee's samples	20.03	
Total expenditures for year 1904-'05	$208.28	208.28
Balance on hand November 14, 1905		$ 77.07
Unpaid dues for 1904-'05		20.00

Respectfully submitted,

Treasurer.

On motion the Convention went into executive session and the following were elected members:

Active:—

F. P. Veitch,
Dr. V. C. Casaburi,
Caspar Drueding,
Wm. A. Fox,
J. H. Russell.

Associates:—

W. A. Rushworth,
G. P. Herndon,
W. A. Mooney,
W. M. Spaulding.

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The executive session was then closed, and the report of the Referee was presented and read.

The meeting adjourned at 12.30 to meet at 2 P. M.

SECOND SESSION.

NOVEMBER 15, 1905.

Meeting was called to order by President Teas at 2 P. M.

On motion, adopted:—

Messrs. Alsop, Haley, and Wilson were appointed a committee to revise by-laws.

A committee of five was appointed to draw up an official method for the association, consisting of Mr. Reed, Mr. Small, Mr. Norris, Mr. Cushing and Mr. Alsop for that committee.

The Referee proceeded with the reading of the report.

After a recess the presentation of papers was in order and Dr. Casaburi read his paper on "A Method for the Detection and Estimation of Soluble Leather in Tan-yard Liquors."

A paper presented by Dr. Louis E. Levi, of the Pfister & Vogel Leather Company, was next read by Mr. Small.

STANDARDIZING COMMERCIAL METHODS.

Gentlemen:

Of all the questions arising during the practice of a technical chemist who is obliged to busy himself with technical as well as analytical work, one of the most important is, what is the method of analysis employed by the chemist to whom the seller submitted the sample? In my practice this question has come up so often that I thought it well to bring the matter up before the Society of Leather Chemists. In buying soaps, hypo, sod oil and many other materials upon a guarantee, *i. e.*, analysis, I found that the results of an analysis vary so often from one accompanying the material, that the Association would greatly benefit all parties concerned by publishing and introducing a standard by which both buyer and seller are protected and all controversy regarding the different analytical methods employed is completely eliminated. Then any variation of results would be only one of careful and accurate

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work on the different samples submitted. The chemical house would then be able to submit its samples of an article for test with the assurance that the standard upon which the commodity was offered will be determined by the same method. The buyer at the same time gains more confidence in the work of the analytical chemist on account of the uniformity of results. In the matter of the examination of sod oils and moellons for water, one chemist is liable to mix his sample with sand and drive off the water at 105 degrees C.—110 degrees C., while the other heats his sample without sand at 115 degrees C. These two methods will give entirely different results, and the consequence is, buyer and seller enter into a lengthy correspondence resulting in a compromise to abide by the analysis of a third party. The report of the Referee chemist shows a different result from either of the others. The buyer and seller accept the analysis of the referee and settle their accounts upon the basis of his analysis, but the confidence in the work of analytical chemists is fast ebbing and the emanations of Radium is totally eclipsed by the verbal emanations of both parties. If the methods of analysis employed in the analysis of technical products cannot be brought down to an exact science as general analytical chemistry, the methods employed should at least be uniform. The case of the matter cited is only one of the many which confront the Trade chemist and help to make his life miserable or interesting according to the view taken. In my opinion the Leather Chemists' Association would do well to take the initiative to bring this matter before the manufacturers and users of their products. Therefore I would suggest to the gentlemen that a committee of analytical chemists be appointed for the standardization of methods for the analysis of materials used in the leather trade.

Discussion followed the reading of this paper, and, on motion, adopted: Messrs. Levi, Yocum and Nichols were appointed a committee to consider the advisability of enlarging the scope of the work of the association in the direction indicated.

A paper by Mr. F. W. Alden on "Chromium and Acid in Single Bath Liquors" was then read.

Mr. A. A. Claflin then read a report on "Acid Determinations" as follows:

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REPORT ON ACID DETERMINATION.

Circumstances which I could not foresee when I took the responsibility of Chairman of this Committee precluded the direction of collaboration work at the time originally planned.

The opinions of the members of this Committee are, I believe, divided, Mr. Small and Mr. Mosbaugh finding that the charcoal method does not give satisfactory results, and Mr. Norris and the Chairman finding that for purposes of tannery control that the charcoal method does give sufficient concordant results.

Mr. Small uses a gelatine precipitation method, titrating with barium hydrate, using tumeric paper as indicator.

No collaborative work was done on a comparison of these methods.

In my own laboratory a considerable number of determinations made by Mr. F. T. Moses, Mr. J. W. Phelan and myself abundantly confirmed the results of the Committee last year, *viz.:* Under certain standard conditions the charcoal method gives concordant results within the limits required for tannery control. The variable factors are amount of iron and other bases in charcoal.

Schuchardt's, which is apparently the best, containing very appreciable amounts of these. The absorption of acid by the charcoal itself, which is apparently in proportion to tannin, coloring matters, etc., in the liquor to be determined, and in the case of acids added to the tan liquors, the purity of the acids themselves.

The standard conditions should be such as to take these into consideration and those laid down by the Committee of last year do this.

For the ordinary strength of liquors in the handlers, results accurate to 1 in the decimal place if 10 cc. of the liquor is taken, made up to 200 cc. and boiled for 5 minutes with Soxhlet reflux condenser with 2 grams charcoal. For liquors high in tannin and low in acid, errors are greater and some other method may be desirable.

Investigation of gelatine method shows that some acid is absorbed by gelatine and errors introduced thus correspond to those of charcoal method for ordinary purposes. The errors of neither of these methods are sufficient to condemn methods.

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The greater simplicity of the charcoal recommends it to the chairman.

For an accurate method and for determination of small amount of acid in liquors of high tannin strength the magnesium method of Kohnstein and Simond is the best. It was suggested by Mr. Small than an improvement in this method might be devised if the magnesia was determined volumetrically in the filtrate.

Numerous methods were tried, and I hope that collaborative work may be done on this line.

The method above all giving the best results is that of Stolba, given in Sutton, page 110.

This is, on the whole, perhaps simpler than the gravimetric determination, although it depends to a large extent on the individual operator,—many of the agricultural analysts preferring the gravimetric method.

The method is more accurate than the previous methods. But we believe it should serve mainly as a check on them.

The variable factor is the solubility of magnesium oxide in the liquors. As a rule this is negligible, but conditions can be conceived where considerable error might be introduced.

The following resolutions, in the name of Dr. Levi were then offered, and, on motion, tabled:

WHEREAS, The American Leather Chemists' Association has no official journal in which the proceedings of the annual meetings are printed, and

WHEREAS, Such a journal is needed to publish the proceedings of the Association and the weekly work of the individual members. Now, therefore, be it

Resolved, That one of the trade journals now being published be selected by vote of this Association as the Official Journal; and be it further

Resolved, That the Secretary be and hereby is instructed to secure the vote of members of this Association by mail. And further, be it

Resolved, That the polls close on the 31st of December, 1905, at 12 o'clock, noon, and that the Secretary of this Association act as teller and the Journal receiving the majority of votes cast, shall be duly designated as the Official Journal for year ending Dec. 31st, 1906.

Meeting then adjourned to meet Thursday morning.

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THIRD SESSION,

THURSDAY MORNING, NOVEMBER 16, 1905, 10 A. M.

The meeting was called to order at 10 o'clock A. M. by the President and immediately went into executive session and elected Mr. T. J. Mosser an active member of the Association.

On resuming the regular session, Mr. Reed exhibited his new form of extractors and read a paper describing them, and giving results obtained by their use.

Mr. Veitch then exhibited and explained the form of extractor in use in his laboratory.

MR. VEITCH. This is the extractor that we have been using in the laboratory of the Department for several years now. I brought it out at St. Louis last fall at the meeting. It is an ordinary condensation Soxhlet tube extractor, and in loading it we place a perforated porcelain disk in the bottom and put the material on wet. After placing in the material and pouring back the first percolations, which are usually very cloudy, we place another porcelain disk on top and set this in a flask, and then you can proceed as desirable, by pouring on hot water or cold water to drip on it from a reservoir. The first solution may be obtained at any desired temperature below boiling and does not need to come under the boiling temperature at all, if the percolation at low heat is continued for a time—400 cc. we usually use. We put 600 cc. in a flask and connect this with a lead pipe condenser and allow to extract at steam heat. As the extraction continues and as the extract becomes concentrated it is removed from the receiving flask added to the previous off portion and the extraction continued with fresh water until complete.

I will say here that this side tube in this particular extractor is rather too small. This extractor holds about 60 grammes. It can be made, of course, of any desired size.

The side tube extractor, I think, is worth mentioning, and I have seen it elsewhere in copper. It is very much more easily handled. I have found it very effective. The results by this extractor have been uniform on all materials that we have tried from sumac to barks and woods. The results have uniformly been higher than with the Proctor form or with the Soxhlet form. If it were used with the Soxhlet, extraction can be made to give as high results as any. It is only a question of continuing it long

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enough and having enough condensation, but it seems to me that the continuous dripping is going to extract the material very rapidly. I have had this made smaller than the old form because I wished to have the benefit of the height of the column. A given amount of water will extract more from a tall column than from a short column.

With regard to the effect of starch. As I say we have tried canaigre in this extractor and I think most people think this is the most difficult to extract on account of the starch, the temperature having to be kept down to about 50 degrees. We tried the Proctor extractor to 50 degrees, and by this boiling temperature we got about 1 per cent. more tannin than we had obtained by the other cold extraction.

With regard to the reds that Mr. Reed spoke of, the reds precipitated in the concentrating solution—I thought we had the same difficulty on sumacs and barks—and we ran duplicate sets, extracting the first off with cold water and extracting a duplicate set with hot water. When we got the results they were practically the same thing within the ordinary range of error, so that with the material we worked our results all seemed to indicate that the reds which had undoubtedly first been precipitated had gone again into solution. We have gotten uniformly high results. So far I have never been able to get any information or data indicating a destruction of tannin during the extracting process. It may take place, but I have no basis on which to determine it, as this boiling temperature has uniformly given us higher results than the cold temperature.

MR. REED. I would like to ask if, in Mr. Veitch's opinion, the continued boiling of the solution from the beginning, practically, of the process until the end of it, does not produce a loss of tannin.

MR. VEITCH. I do not know. The only thing I can say is we have gotten higher results in this way. We have no absolute data showing the destruction. You are getting lower results by your old percolator, but whether it is due to a destruction of tannin or to a failure to extract tannin I do not know.

MR. REED. It is the high non-tannin in the old method that produced the low tannin.

MR. VEITCH. We have not gotten that even with the old extractor. Our results have not been proportionately higher, but

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they have been higher with this extractor than with the old. We have been using the old extractor for the past two months. I think that we are criticising the extractor rather than the particular size of the extractor. If it were modified, if it were brought down smaller, so that we could boil such a solution and concentrate it down, I believe I can get much better results with a smaller extractor. The other day we ran some by three methods, by the Proctor method, by this condensation extractor and with the Soxhlet extractor. With the Proctor to get a litre, I think it took us about eight hours. We ran with this condensation extractor about the same length of time and then stopped. Then we ran it for twenty hours and stopped that, and found with this extractor running just the same length of time as the Proctor we obtained higher results right straight through than we did with the Proctor. They were not as high as with condensation for twenty hours. Another point is, this apparatus costs, I think, about $40 per dozen. The simplicity of it makes it, of course, cheaper.

MR. REED. There is one point. I do not know as you understand my point of starch in sumac. It was not in boiling any given fraction, that the starch actually produced any loss.

MR. VEITCH. Yes, that is the point that is very well brought out. I saw your point on that. It has been considered generally that starch does do that. At first it was considered that starch held back the extraction of tannin, and it may do so, but I think it can be removed by keeping on the extraction. But our results do not indicate it even on canaigre. Our results are higher by condensation even if we pour off the last portion. It is higher than where we get results with the Soxhlet extractor. We get at these things by difference. We have no positive way of determining this.

MR. REED. Mr. Alsop has done some work on extraction. I would like to have his views on the relative value of the condensation extractor as compared with removing the extraction by percolation.

MR. ALSOP. I believe percolation is the best way to extract. I do not think there is any doubt that the old form of extraction destroys tannin. I think by percolation, if it is properly done, you can get as much tannin as you can in any other way.

MR. VEITCH. To what volume ?

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MR. ALSOP, Two litres.

MR. VEITCH. Are you going to concentrate that ?

MR. ALSOP. I will analyze it as it is.

MR. VEITCH. And will you have the strength desired ?

MR. ALSOP. What strength ?

MR. VEITCH. Thirty-five or .45 grams tannin per 100 cc.

MR. ALSOP. No, you will not get that. In the siphon Soxhlet, by long continued extraction, you can get possibly as much as you can get by any extraction.

MR. VEITCH. I do not doubt that, with figures obtained by the complete extraction, but we cannot get anywhere near in the tannery what we get in the extractor where we make a complete extraction.

MR. ALSOP. One reason I think a percolator is a good way to extract is because it is under somewhat the same condition as we have in the tannery.

MR. VEITCH. If you can get it all, it is all right. I have never been able to do that.

MR. ALSOP. I think I can.

MR. SMALL. In connection with the question of boiling solution in the Soxhlet, I have made some experiments which are not complete and not conclusive, and yet they throw some light on the matter. I took a sample of extract and divided it in exactly equal proportions, and to two of them I added enough water to make it up to 225 cc. and to another portion to make it 450 cc., making up as you do an ordinary solution for analysis. The two first solutions I put into a flask and connected it up with return condenser and then I boiled the solution just as near as I could under the conditions which would obtain in the ordinary Soxhlet extractor. I boiled for nine hours and at the end of that time I made up my solutions. One of them was of 450 and the other we made up to 450 and allowed them to cool over night. I analyzed the three liquors I obtained in this way. The original extract corresponded to the liquors made for analysis as follows:

	225 cc. boiled 9 hours.	450 cc. boiled 9 hours.	Regular Analysis.
Total Solids	39.99	40.07	40.19
Soluble Solids	39.26	39.48	39.84
Non-Tannins	14.86	14.76	14.13
Tannin	24.50	24.72	25.71
Reds	.63	.59	.35

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On concentration solution there was an apparently greater destruction of tannin.

MR. YOCUM. Is there a loss from the boiling ? Is there a loss of the tannin affected by the conversion into non-tannin ?

MR SMALL. That I am not prepared to say, but as analyzed by the hide powder method, the prolonged boiling does show a distinct loss in tannin.

MR REED. This bears out the results I got in comparing the Soxhlet with this form of extractor. It is in the non-tannin.

MR. VEITCH. The results would certainly indicate some destruction of tannin. Taking that as a basis, that is very well brought out there I think, but it evidently is not a very large destruction. It was about one-half per cent., was it not ?

MR. REED. Over one per cent.

MR. YOCUM. All sorts of raw material must be considered. For instance, you take and boil up hemlock bark, one of whose characteristics is to change into reds, you get a great deal less in the old Koch extraction than you would by the use of wood in that extractor. At the same time it probably gives a greater amount of tannin than you would get in the old Koch extractor. It seems to me that extraction by means of the Proctor extractor is not as complete, yet the facts are, you find just as much tannin as you will from the Koch and yet you know it is not complete. You cannot carry it to an absolutely extracted condition. Mr. Reed refers to some condition in which the non-tannins indicate a loss of tannin. I have no doubt in my mind but my experience has always been that where the extraction was unusually high the tannin is not indicated by that at all because the tannin stays in the raw material and does not come out in the solution at all.

MR. REED. I do not see how that coincides with Mr. Small's statement about making a solution of the extract and boiling with the condenser, whereby he says he had a distinct loss of 1½ per cent.

MR. ALSOP. I think the increase of non-tannin is due largely to something dissolved from the wood and is not made. I think in the Soxhlet extractor it is caused largely.

PRESIDENT. That is in the case of chestnut wood ?

MR. ALSOP. Yes.

THE PRESIDENT. Would you say that about bark too ?

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MR. ALSOP. Yes. If you make that with a percolator and carry it on for quite a while, you will get an extraction that has a small amount of solids. If you continue, I think you get the same results and you bring up your non-tannin.

MR. REED. I think what Mr. Alsop says is true, but nevertheless I do not think that answers the results Mr. Small obtains. I think there is some loss.

MR. ALSOP. I think there is.

MR. VEITCH. Did you state the kind ?

MR. SMALL. It was a chestnut wood extract and the flask was Jena glass or non-sol.

MR. VEITCH. I do not think it was a conclusive test.

MR. YOCUM. I am certain the longer you extract a wood or a bark the higher your non-tannin will be. If that is not conclusive evidence, that beginning at a point where you have the highest percentage of tannins, where you have gotten as far as the Koch extractor goes, and continuing that with another relay of water, you will continue to get non-tannins. That I know.

MR. VEITCH. I do not think that the continued extraction of non-tannins in conjunction with tannins, which also continue to be extracted, is any proof at all of the destruction of tannin. You may continue to get non-tannin, but you will also continue to get tannin provided the extraction is not complete.

MR YOCUM. But if you have extractions for twelve or fourteen hours and then again for twenty-four, you will only get so much as the natural error in the analysis would be.

MR. VEITCH. I do not agree with that. I can get a very heavy precipitate with gelatine, and experiments of that kind do not establish the matter at all, like experiments such as Mr. Small has conducted. That is definite information.

MR. CLAFLIN. I think if Mr. Small had made his experiments on the hemlock extract he would have shown the destruction of tannin by boiling much more noticeably than with the chestnut wood. I think hemlock bark, and a good many other tannins, are more susceptible to the influence of boiling than others, and I think if some experiments were made on that line—I think there are published results—I think Proctor has some,and I know that the statement is so general that there is a loss that I think it will be shown very evident by work done on the line Mr. Small has

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worked on . I think with regard to the increase of non-tannins if you take any wood and keep on extracting it would give a gradual decomposition. The point is to stop when you get no further indication of tannin material, and it seems to show by the Soxhlet method that you get a higher tannin as well as non-tannin.

MR. REED. I would like to inquire here why, with an extractor like this, when you compare it to the Soxhlet and calculate your analysis to the same soluble solids, you invariably get higher tannin,—lower non-tannins and higher tannins. Why is that so, if there is not a destruction of tannin ? What has become of it ?

MR. CARR. I would like to inquire of Mr. Reed whether he has worked on progressively increasing quantities of material, using more water.

MR. REED. No, I have not.

MR. CARR. Mr. Wilson's experience was that in case where he started with about 20 grams and went up 10 grams until he reached as much as his cup would hold, my recollection of his results was that the tannins seemed to strike a pretty fair balance. The non-tannins showed a constantly increasing percentage until he struck a level of about 100 grams, where both showed more constancy than they had in any other increase of 10 grams. I would like to inquire of the President whether in the same results he gave he believes that the new extractor gives a larger figure than the old. Is it your general impression that the new extractor gives a better range of figures ?

PRESIDENT. I have only done a little work on that. I got switched off on the old Soxhlet, but I did some of the samples. I got results higher in tannins, but not very much lower non-tannins.

MR. YOCUM. I do not think there is any question but that there is destruction of tannins by boiling. The only question I brought out was whether the destruction was due to the non-tannins or to the getting into the solution of some of the wood substance. I believe the increase of non-tannins is largely due to the going into solution of some of the wood substance. Undoubtedly it is not wholly due to this, but to the fact that with sumac and certain other tanning materials the boiling temperature will have a distinct effect upon the percentages found, is the best evidence that it does occur.

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PRESIDENT. Personally, I believe it is due to a more finished extraction.

MR. YOCUM. That is what I believe.

MR. REED. Relative to what Mr. Carr has stated, it occurs in last year's report. His results are not uniform with varying qualities. He used 20 grams, 40, 60, 100, 120 and 140. His results run from 2.61 per cent. to 2.40 per cent. that is the constant tannin. The soluble solids run from 5.53 per cent. to 4.60 per cent. The non-tannins run from 2.92 per cent. to 2.17 per cent.

MR. VEITCH. In other words, the difference was in the soluble solids.

MR. CARR. The soluble solids. You can look at it the other way and say the totals were reduced as the amount of material increased, and as the solution increased in strength, his volume remains the same but his non-tannin is increased.

MR. VEITCH. The non-tannin increases ?

MR. REED. It seems to me there are one or two points to be questioned. I presume, of course, the hide powder was in ratio to the amount of material extracted.

MR. WILSON. Yes, sir.

MR. REED. Then they were all made up to 1 litre ?

MR. WILSON. Yes. The thing I was trying to get at was that some use 20 grams and some 40 and up to 120, and what I did was to show that somewhere along the line was a point of stability, and there should be some certain amount defined for use in all cases.

MR. REED. If the extraction was complete in every instance, you should have total solids equal in every case. If that was the case with the total solids, the difference in the soluble solids must show that there was a difference in the reds. If you keep a constant solid and a constantly decreasing soluble solid, there must be a constantly increasing amount of insoluble, which I think bears out my remarks.

PRESIDENT. Do you think you can place any reliance on the total solids in an extraction of bark ? Are there not more or less fine particles of bark in which reds come through ? I do not think you can place any reliance on them.

A MEMBER. The point I wish to make is, it may be in the analysis that the difference is shown. If you have any extract and

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make up a solution twice as strong as another solution, you will get different results.

MR. CARR. It seems to me if we are to ascertain whether bark is present in the solution, the most direct and simple way is to heat the solution and find out, and not introduce a lot of unknown factors, any one of which may confuse the entire calculation.

MR. SMALL. I would like to say that there is at least an even chance that there is an error in the analysis brought out in this way. I cannot speak so positively in regard to extract solutions, as I can with liquors, but I have worked with the point in view, that if you have a solution made up and analyze it with a certain amount of hide powder, and then take that same raw material and make up the solution with half the quantity you did before, and use half the hide powder, the results are not just half what they were in the other case. If your solutions are not the same you cannot get corresponding results by diminishing your hide powder at the same ratio that you diminished your solution. If those analyses were made by proportioning the hide powder to the strength of the solution, the results are not comparable.

MR. WILSON. I remember at the time those figures were made I made them directly for Mr. Craighill. We were talking about the matter and after I tabulated them I wrote and told him that in the first instance where a small quantity was used it seemed to show that there was substances that were soluble in a weak solution, which were really chargeable in the soluble solids. Where you produce a higher concentration the reds come out. I wrote that to him, but did not include it in the paper.

MR. VEITCH. I hope Mr. Small will continue his investigation with other extracts. It seems to me that is the proper way to approach the problem.

PRESIDENT. Is there anything further on this ? I do not know that there is anything to be gained in view of the lack of collaborative detail.

MR. WILSON then took the chair and Mr. Teas exhibited and explained his new form of extractor.

MR. HALEY. I would like to ask some of the chemists here what is the practical application that they would place upon these different forms of extractors and extraction in the actual tannery leach house. Whether the same principles will hold true in the

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leach house that they are claiming for these different forms of extractors ?

MR. REED. I think it might be answered in a measure by another question. Have we in the past with our old form Soxhlet gotten the amount of tannin out of fresh material that can be removed by extraction or by leaching ? I do not think we have. I think we have in the past gotten more tannin in the leach house than we have in the soxhlet extractor.

MR. YOCUM. My experience with bark extraction is that the analysis always indicates a higher percentage than the practice, and in woods I understand it is the reverse, so if you want to get something that actually represents practice you will have to have two methods.

MR. VEITCH. It seems to me it all comes back to the experience we have in many kinds of chemical work. It is whether you want to get the total amount of tannin or the available amount—what the tannery will get out under the best conditions of operation. It seems to me that there are two entirely different things. It seems to me that one is a chemical method and the other one available for tannery practice, that is, the tannin that could be gotten out under the best conditions of extraction.

MR. SMALL. I must beg leave to differ. I do not think there is any necessity for a method for determining available tannin. We want to get at total figures. The old form has not given us total figures. We have not been able to get total figures. We have handled our extraction so that the results we obtained in analysis did not represent actually the amount of material there was in the substance we have extracted. We want to get some absolute means of determining how much tanning material is obtained by the combination with hide powder, and that is the thing we want to get at, not as to whether a man in his leach house can get that much out of it or can get only half of it out. I don't see that it makes any difference. The leaching done in the leach houses now is a distinctly different proposition than it was ten years ago, and the man who is conducting his business on that method would probably go out of business very soon.

MR. REED. I want to say that if we are going to get comparative results on extraction there is only one way to get them, and that is by getting as near an approximate extraction as possible.

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We cannot get them under any other method, in my judgment.

MR. YOCUM. In connection with your statement a few minutes ago in reference to the practical question on method of analysis, the conclusion you draw must be that the total amount found in the analysis of the wood against the total amount of tannin contained in the extract, plus that which is left in the spent wood—if the two figures do not agree—you do not mean to say that the actual result in extract was greater than that which you found in the wood. You had to make an analysis of the spent wood before you found a greater quantity.

MR. REED. No, I can take extract and find out the percentage of tannin of the extract and from it figure back to the weight of tannin contents, which I could not actually prove if I had extracted this in the laboratory in the old form of Soxhlet. The trouble has been we have not gotten out all the tannin that is in there.

MR. YOCUM. I understand your point, but how do you prove it? Do you find that you can remove the greater sum than you find in the laboratory?

A MEMBER. You find in a certain weight of wood 100 pounds of tannin and it turns back 110 pounds, not considering the spent wood at all.

A MEMBER. Do you confine that to Quebracho wood?

MR. REED. Yes, I confine it to Quebracho wood, for that is the one I tried.

MR. VEITCH. I should like to say that personally I believe in the method for the total. I can see particular conditions which would make it desirable to know hom much could be gotten under best methods of factory work.

MR. SMALL. That would be all right if the best methods in factory work were a constant quantity, but they keep varying all the time.

PRESIDENT PRO TEM. Any further points to bring up?

MR. HALEY. I would like some expression of opinion upon the point I asked some time ago. What is the most practical application of the point as applies to the leach house? We should get some points as to the best methods in practice from what we have found out in theory. Is it best to have a ten-foot leach or a four-foot leach?

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MR. SMALL. I think Mr. Haley wants to get us in trouble with the concern we represent.

MR. YOCUM. It would appear from what Mr. Reed says that you will have to come back to extractors.

MR. REED. It seems to me that it certainly opens up a field for the extract people. It gives them an idea as to the degree and method of extraction and temperature. I think the conditions are so varying that it would be impossible to lay down any hard and fast rule. There comes a point in the actual extraction which is not reached in laboratory extraction. In actual extraction it would not pay us to leach beyond a certain point. The cost of evaporating a solution containing so small a percentage of tannin would be greater than the value of the extract you obtained. That is not so in laboratory practice.

MR. CLAFLIN. I was much surprised to hear Mr. Reed say that he got a bigger amount of tannin than he got from the old soxhlet extractor. I have never had any experience with Quebracho, but in every other work I have tried the result has been much higher than I could get with actual practice. I suppose with quebracho he finds some spent tannin which seems to show that the Soxhlet process is much farther away than any of the improved apparatus show. And it almost seems to me that there must be some trouble in the calculating of the practical results. I know in many cases I have heard of bigger yields being made in the factory than in the laboratory, but on investigation they have never been borne out. I cannot concede that they make an error in the amount of wood, but when they use one quantity one day and next day use another it makes a very great difference. I know in the distillation of wood that I have had to do with lately they got bigger results in actual work than in the laboratory work. I don't believe with the Quebracho wood that he can extract any quantity and I think it must be that the methods of figuring out the yield are not accurate and, in fact, it is not easy to figure out a correct yield.

MR. REED. I will say that the conclusion I spoke of is based on actual weighing of the wood.

MR. CARR. It seems to me we are getting in deep water. The question of the application of laboratory results brings in a number of different factors. Take, for instance, Quebracho wood. It

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contains certain soluble matters, as you increase the pressure in autoclaves, as compared with extraction under atmospheric pressure, so that the analytical method may be an entirely improper one. Another thing is in the proposition to advance a set of rules for the manufacturers of leach houses, for instance, and we begin to get into deep water. What is good on hemlock bark may not be good on chestnut wood or oak bark. It would seem to me that the statement of Mr. Reed is pretty near right. What we should seek would be the absolute contents in any particular material as experienced by the extraction under given conditions. If it is possible for the manufacturer to approach this condition, either by one apparatus or another, that is up to him.

MR. YOCUM. Do you find that the extraction by percolation gives you anything concordant with your practice? You speak of the Soxhlet being not in accordance with practice. Is the percolation method?

MR. REED. I have not used it on the Quebracho.

MR. YOCUM. Of course, if he uses the percolation method he probably has found the same results as you have. Most of the tanners of the United States, not extract manufacturers, are users of bark and conditions found in leaching bark are different from those of leaching wood.

I will venture to assert that they do not obtain 8 per cent. of bark as soluble tannin in their liquors and invariably the percentage found in the soxhlet will run higher than that. It will run to 10 or 12 per cent.

MR. HALEY. I can second what Mr. Yocum says in relation to hemlock bark and our bark and it seems to me that in the analysis of spent tan—we do not as chemists give the tanner the actual amount of available tannin left in the spent tan when we send in an analysis. We take a sample of spent tan and dry it and grind it. It is not a practical thing for the tanner to take this spent tan and dry it and then leach it.

PRESIDENT. I should think that method would be open to a great deal of error. I should think it would be better to take all the samples and eliminate all the error and grind them to the same degree of fineness and then sample them and a man establish his own standard as to what point he can reach with his preparation,

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and all above that which an analysis shows is due to his carelessness or laxity.

MR. REED. Let us suppose that we take a preparation as it comes from the tannery and extract it. Where are we going to stop? We cannot get it out with any method.

MR. HALEY. You will stop at the same place you stop now.

MR. REED. I think there will be a great variety of results.

MR. CARR. I think any commercial chemist has had poured upon his table various samples from various tanneries—has probably been embarrassed by a variety of preparations. From one tanner it is evident from the material spread upon his desk that he is getting more tannin out of his bark. The condition of material is absolutely necessary for a given leach house result. If the chemist is going to do anything more than to take the samples as he receives them—he will have in one material that will not pass through more than a mesh one inch square and another ten to the inch.

MR. HALEY. I would not analyze the sample.

MR. SMALL. I should say it would be just as well to have some man who can run a pair of railroad scales—there is no use for any chemist.

MR. REED. You would have to make the analysis for that tanner first in order to prove to him that he had to make his preparation fine.

MR. YOCUM. That is the method that gives you the ultimate. It seems to me that is what we are here for, to find out what method gives you the ultimate. Are the recommendations to guide us in the referee's report relative to that?

The President again takes the Chair.

PRESIDENT. Is the Committee on the Official Method ready to report?

MR. REED. I am the Chairman. The Committee met last evening and this morning to work this out. They have altered the recommendations in a number of instances. I will read you their conclusions and I would suggest that each item be discussed as it is brought out.

"Crude Materials. 1. Moisture Determination. Upon receipt of sample grind promptly and dry 10 grams in the manner

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and for the period specified for evaporation and drying in extract analysis."

MR. SMALL. I would suggest that only one article be read at a time and that that article be accepted or rejected by the members before passing on to the next.

The motion was seconded.

MR. HALEY. What vote of the members present is required to adopt it is official?

PRESIDENT. A majority of the active members present. Any further remark?

The motion was put to vote and passed.

PRESIDENT. I might say in connection with this that I judge from my short attendance at the meeting of the Committee that some of these points led to an interminable discussion and I think on those particular points if the Committee would explain at the time what appeared to be the arguments *pro* and *con* it might save us a lot of discussion here.

MR. REED. In order to bring that out I would read first the method as recommended by the referee yesterday, as read yesterday, and then the alteration in it, and the discussion that led to the alteration. Is that your idea?

PRESIDENT. Yes.

The various articles in the proposed Official Method were then successively read, discussed, and adopted as revised.

Article 4, reading:—(4) Extraction of Sample. Extraction shall be conducted in a form of apparatus that permits the removal of the extractive solution from the influences of sustained high temperature, and shall be continued till a portion tested with gelatine salt solution fails to give a precipitate. A least 400 cc. of the first portions of extractive solution should be removed and not subjected to further heating.

"In case of finely ground material, it is permissible to use glass sand, or thin layer of cotton, to assist extraction," brought out the following discussion:—

MR. YOCUM. I understand from this that any apparatus permitting the removal of the first portions will be all right, but where and when are we going to stop the extraction? I do not see there anything indicating the point to stop at, either time or temperature or any reaction that is feasible. It says "shall be

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continued till a portion tested with gelatine salts solution fails to give a precipitate." That is to my mind a very elastic limit.

MR. SMALL. In reply to Mr. Yocum I would say that as I remarked earlier, a gelatine salt solution will with certainty detect one part of tannin in 30,000.

MR. YOCUM. But how do you show it?

MR. SMALL. Take 10 cc. and add one drop of your liquor, and if there is tannin there,—if there is tannin to the amount of one part in 30,000, you will get a distinct precipitate. You can detect one part in 30,000 without any hesitation whatever.

MR. YOCUM. Could you reach a point when you could get your solution off and not indicate any tannin?

MR. SMALL. Yes, I have succeeded in doing it.

MR. YOCUM. How many times out of a hundred?

MR. SMALL. If I want to do it, I do it right along.

MR. YOCUM. I want to know how much work it entails.

MR. SMALL. It can be done in the usual working day without any difficulty.

MR. YOCUM. How about the Proctor extractor?

MR. SMALL. I do not think the ordinary extractor is as well fitted as Mr. Reed's extractor. The one I use is one made after the idea suggested by Mr. Forbes along in '97 or '98. He published an article on the extraction of different tannin materials in which he advocated the form of using the Soxhlet in the water-bath, and that is the form I use where I use the percolator. In my ordinary work I use the Soxhlet, but I will confess that it does not represent the full tanning content.

MR. YOCUM. May I ask why you use the Soxhlet in your usual work and the percolator in your accurate work. Because the percolator is more elaborate to operate?

MR. SMALL. Yes, the percolator does require some attention. The Soxhlet does not.

MR. YOCUM. What difference in per cent. do you find?

MR. SMALL. To answer that, I would refer you to Mr. Reed's table.

MR. YOCUM. I was asking you from your experience.

MR. SMALL. It will run from ½ per cent. to 2 per cent. and more.

MR. YOCUM. On the content material?

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MR. SMALL. Yes.

MR. YOCUM. On spent bark?

MR. SMALL. It would be a fraction of a per cent.—just how large a fraction I could not say. The objection to using the percolator is that I have found that it ordinarily requires more time and attention than I can give to it. As my extractions have been conducted, nine hours would not be sufficient to give a complete extraction of a good many materials. With some it will be enough and with others it will not. There is no reason why any material cannot be extracted so completely that it will fill the requirements by this method.

MR. YOCUM. I understand there would be no reason why you could not do it, but the idea is whether the increase in tannins justifies the amount of work it entails. For instance, Mr. Teas and Mr. Alsop make a great number of spent bark analyses and it would take from one to three men to examine them. We do not want a lot of furbelows and frills. We want a practical method.

MR. SMALL. That is true, but it can be readily seen by drawing off a little. It shows when your percolation is practically complete. Suppose you have a number,—it will take not more than ten minutes to examine the whole. Unless some method is arrived at which is absolute, there is no hope of getting uniformity.

MR. VEITCH. As I said some time ago, the test for tannin is, of course, influenced by the concentration. Now in the old form of Proctor extractor or in fact any percolation method, there will come a time when you will no longer get the test for tannin and yet if you continue that extraction in an extractor that extracts tannin in a small volume of water,—in other words, give us a much more concentrated extract, you will get a heavy precipitate of tannin. That is the only point I see. It leaves a great deal of laxity. One man using a percolator would stop because he found no tannin. Another using an apparatus like we use would have to continue three or four days before he got to a point where he would not get any more. I do not know that you could guard against that, however.

MR. SMALL. The trouble is, if you specify too closely, you have to specify a different method for every tannin material and you

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would complicate it so as to have the frills and furbelows Mr. Yocum objects to.

MR. VEITCH. That is true, but the man who will use the old percolator form will stop at a litre or two litres and get no reaction. I never stop at one litre, but I have stopped at two and never got a reaction, but I can put it in this extractor and get 2 per cent. or 3 per cent. more. It is a question of the concentration of the solution you are testing that will control.

MR. CLAFLIN. I would like to ask about the thin layer of cotton. I have seen it put in for a good while. Does not filter paper have an absorption effect on the tannin and why not cotton?

MR. REED. Because you can wash it out with water.

MR. CLAFLIN. Will that remove the tannin?

MR. REED. Yes. sir.

MR. HALEY. I do not believe it does because you can make a dye test on cotton to obtain its color and I have never seen that color removed from cotton with hot water.

MR. REED. How do you make the dye test?

MR. HALEY. With a mordant.

MR. YOCUM. I move the clause be adopted as it reads.

The motion was seconded, put to vote, and passed.

MR. REED. "(5) Analysis. After extraction and dilution, solutions must be heated to 80 degrees C. and analysis conducted as per Official Method for Extracts. In case of weaker dilutions than the Official Method specifies, the amount of hide-powder must be reduced in proportion to the reduction of tannin."

"Ten grams of the air-dried sample should be dried, as in Paragraph 2, to determine moisture content of the portion extracted, and the analysis calculated and reported upon a 'dry' basis."

A MEMBER. I move its adoption.

MR. CARR. Is that point to call attention to the necessity for filtering the solutions other than from the extractions of woods and other fine materials? Is it necessary for the method to be specific on that point?

THE PRESIDENT. If cotton is used, it will not.

MR. CARR. Possibly new workers should have specification. Unless the fibre is eliminated promptly after the liquor is extracted, the red determination is of course valueless, and it would

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seem at that point it might be well to specify that it be at a certain temperature.

MR. REED. It shall be conducted as per Official Methods for Extracts.

MR. CARR. But that does not cover the point. After it is cleared of its fibre, it must be filtered.

MR. ALSOP. He means, we have not made any provision to get rid of the fine bark.

MR. YOCUM. There is no provision made in the Official Method because it is not supposed to have any bark.

MEMBER. Let it stand ten minutes, perhaps.

MR. REED. No, half an hour perhaps would be better.

MR. YOCUM. I would want it to stand for a long time because very fine particles of bark will float for a long time.

A MEMBER. You cannot separate fine particles of bark from the reds.

MR. CARR. Some of us might try the extraction at a relatively low degree, perhaps 40 degrees, and filter it through the cotton; others might take it up promptly and filter it at 80.

THE PRESIDENT. According to the method, there has been at least 400 cc. removed. If they want it through paper, they have to heat it up.

MR. CARR. This is the point I want to make.

THE PRESIDENT. Why not substitute a cheese cloth?

MR. REED. I do not see the necessity.

THE PRESIDENT. With the fine preparation, the reds mean nothing at all.

MR. WILSON. If you want the total solids, it must be separated.

MR. VEITCH. A very good thing is silk bolting cloth such as they use in mills. You can get it most any size mesh you desire, and you could get something of that kind. Of course silk will absorb some tannin, but you would only use a small piece.

MR. REED. My practice has been after making and mixing my solution, to pour into a cylinder and allow it to stand one hour. I find I have no trouble with the fibres. They settle out sufficiently. I work a great deal with fine preparations.

A MEMBER. How about reds coming down? It strikes me an hour is rather long. I think that is probably the best method.

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THE PRESIDENT. We allow it to stand in our laboratory a half hour.

MR. VEITCH. Would it not be as well to refer this to the Referee and ask him for some results next year?

MR. HALEY. I don't think it requires a year's study. We have all had experience along these lines. We use cheese cloths and take out the bark. It is absolutely necessary in some tannin materials to ascertain the amount of soluble matter.

MR. ALSOP. I think if we are going to use the material ground to pass a 20-inch-mesh, we should specify that and it is practically what anybody would do.

MR. SMALL. I agree with Mr. Alsop. I think there is no method of eliminating the reds if you allow it to go on. Either you will get your reds mixed with the material, or if it is arranged so that the liquor is free from the dust, you will save that trouble and you can get an accurate determination for the reds.

MR. CARR. Don't we run into trouble there? Does not the method leave it optional whether the operator shall use a percolation method or a filtering method? If he uses the filtering, the body of material rises to the top of the siphon, particularly while the material is new and it is likely that the cotton covering will be lifted or disarranged. I have seen Mr. Wilson's cylinders standing with the extracted liquors. I think his practice is to take them off from the frame and let his cylinders stand until morning. I have noticed cylinders standing with quantities in the bottom two inches deep. If you are going to leave the point open so that either method can be used, we are liable to run into trouble with the disarrangement of material on the cotton filter and without exception you will find occasionally liquors mixed with reds, and it should not be left open.

MR. SMALL. I think that is something the individual operator must look after, and so far as the rising of the cotton layer is concerned, that can be obviated by having a layer of sand on it.

Q. Has that method been prescribed?

A. No, but it is utterly impossible to prescribe every method. Something should be left for the operator.

MR. CARR. Occasionally I get a letter from a new man in the business seeking for information. It develops upon investigation that he has had copies of the method and studied them with

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avidity and his letter indicates familiarity, but he says, "I do not understand this or that," and if there is a commercial value to be ascertained of the materials containing reds, why not make an effort to find a method on that point? Why leave it open to a possible contingency that may lead we know not where. It seems to me if we have had experience of fibre coming into the extraction materials, that ought to be enough. It occurs. It may occur with care and manipulation, but it would not be fair to leave a misunderstanding with a new man who will take the method in good faith.

On motion, adopted.

The Secretary was instructed to prepare a clause covering the point under discussion, and insert in the article.

Articles 6, 7, 8, 9 and 10 were adopted as read.

MR. REED. We come now to the Analysis of Liquors.

"11. Dilution. Liquors must be diluted for analysis so as to give as nearly as possible 0.7 gram solids per 100 cc. of solution."

THE PRESIDENT. I would suggest that immediately on the completion of the reading of the paragrph some member of the committee should move its adoption and bring it before the Association in that way.

MR. SMALL. I move that paragraph be accepted as read.

MR. YOCUM. Why in one case do you make it .35 to .45 grams?

THE PRESIDENT. In one case it is .35 to .45 grams tannin, and in the other case .7 grams solids.

MR. REED. Were you here yesterday? That is in the work of this year. There were three methods tried. From the results of the collaborators, the Referee could only consistently recommend such a method as here given.

MR. YOCUM. The reason I asked this is that at previous meetings I have always felt that the method should use grams of solids and not grams of tannin, and I wondered why you came back to the former method in this particular.

MR. ALSOP. We have not come back to it, except in a certain manner, as you will see further down.

MR. SMALL. I want to go on record as being fundamentally opposed to the use of this method of mixing liquors for analysis. I said so at the time in my report and to various members of the committee, and I do not want to burden this gathering with it,

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but I do not want to be understood as believing this is the most desirable method. I am perfectly willing to give my grounds to anybody individually. I want to go on file as opposed to it.

MR. VEITCH. I think the members of the Association, except the committee probably, are voting on many of these changes somewhat in the dark, and I rise at this time to ask that next year the Referee, if possible, will have his paper so arranged that the tables can be seen, because then we will know how we are voting. Perhaps, some of us would vote differently if we saw the figures.

MR. REED. I will give you a copy if you wish it.

MR. VEITCH. There is hardly time now.

MR. ALSOP. I should think it might be as well to say that the committee decided to make as an experimental method to work on next time, the method proposed by Mr. Small.

THE PRESIDENT. That comes up later.

MR. YOCUM. I can understand the necessity of what the committee has done. Since Mr. Alsop has told me of the reduction of the amount of hide, it seems to me that covers the original objections that were made to the proposition that Mr. Small presents.

The motion was put to vote and passed.

MR. REED. I would suggest that we complete the reading of this method and then go back, as you can't separate one portion and understand part of it at a time.

MR. YOCUM. I suggest that you read 12, 13 and 14, and have them adopted together.

MR. REED. "(12). Total Solids. To be determined as in Extract Analysis.

"(13). Soluble Solids. To be determined as in Extract Analysis.

"(14). Non-tannins. To be determined by shaking 200 cc. of solution with an amount of wet chromed hide powder, containing 70 per cent. to 75 per cent. moisture, corresponding to an amount of dry hide powder shown in the following table.

"Tannin range per 100 cc.	Dry Hide Powder per 200 cc.
.35—.45 grams.	8—10 grams.
.25—.35 grams.	5— 8 grams.
.15—.25 grams.	2— 5 grams.
.00—.15 grams.	0— 2 grams.

"100 cc. must be evaporated as in Extract Analysis."

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MR. YOCUM. I move that Sections 12, 13 and 14 be adopted as read.

THE PRESIDENT. Any remarks.

MR. VEITCH. May I inquire why the amount of hide here is stated as 8 to 10, while in extract it is from 12 to 13 grams.

MR. REED. It is found by any one who has had much experience, and those who have had a great deal more experience than the Referee, that the amount of acid in the liquor has its influence so that so large an amount of hide powder used in the extract analysis is not only unnecessary, but unwise on account of its absorption.

MR. ALSOP. I have a table here of some analysis which would seem to show—in this case, 7½ grams of hide powder was just as good as 10 grams. If the members would like to look over this, I will be glad to have them see it. I do not think there is any doubt that a less amount can be used just as well.

MR. REED. Mr. Teas called attention to the fact that nothing is said relative to the time of shaking.

MR. VEITCH. I move that it be inserted "Shake 10 minutes."

MR. YOCUM. Why was it not made half an hour?

MR. ALSOP. It seems to be the opinion of the Association that it is necessary to shake extracts 10 minutes. I do not think it is necessary to shake extracts 10 minutes. They might as well shake the liquor also.

THE PRESIDENT. Mr. Veitch, would you change your motion to the form of an amendment and move that the original amendment be amended?

MR. VEITCH. I would move that the same as in extract analysis be appended, just simply to get it complete.

MR. SMALL. You offer that as an amendment to the motion before the House?

MR. VEITCH. I do. That makes it uniform.

The motion was seconded, put to vote, and passed.

The original motion was put to vote and passed.

MR. REED. In connection with this, the committee recommended as an experimental method, the method suggested by Mr. Small. The experimental methods this year mean just what they say—that they are not provisional methods and under no circum-

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stances can the analysis be reported upon by an experimental method.

MR. YOCUM. The experimental method would be for the use of the next year's Referee?

MR. REED. Yes.

MR. YOCUM. It would be understood that that is what it means?

MR. REED. Yes.

MR. REED. I would insert under paragraph 14, calling it A, Mr. Small's suggested method and have it put in as an experimental method.

THE PRESIDENT. But not as a provisional method?

MR. REED. Yes.

MR. VEITCH. Why burden our methods with a method we do not propose to use at all, except for experimental purposes?

MR. ALSOP. I think it is wise to do that so as to bring the matter before the members so if they wish to do any experimenting upon it themselves they can do it.

MR. VEITCH. They can do it without its being in the method.

MR. YOCUM. It seems to me if it were in the proceedings it would be brought before the members quite as well as leaving it where it is. It seems to me if it were incorporated as part of the method it would be difficult to enforce the sentiments that you want to because by incorporating in this method, an experimental method might mean anything in principle.

THE PRESIDENT. It is difficult to define it here.

MR. YOCUM. Yes, let us discuss it outside of the method. It can be brought before the members just as well.

MR. ALSOP. It is before the House?

THE PRESIDENT. No, it has not been read yet.

MR. ALSOP. I move that the experimental methods be put at the end of the official method with a distinct heading that they are not to be used as reporting analyses under the official method. I believe in having them together.

THE PRESIDENT. Does this apply to all experimental methods added to the end of official methods with a distinct heading?

MR. CARR. I amend the motion—that the Secretary shall prepare the heading to append to the official methods, and that the

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heading shall be passed upon by the Association in order that it shall be perfectly clear.

MR. YOCUM. Do I understand that the method shall be kept in this? Will the experimental part be presented to the A. O. A. C.?

MR. REED. No, you could not very well. You see, they have a system over there of provisional methods. I have no right to present that.

THE PRESIDENT. Is there any second to his cross amendment?

MR. SMALL. I second it.

MR. PRESIDENT. Will you accept that, Mr. Alsop?

MR. ALSOP. Yes.

THE PRESIDENT. Then we will vote on the motion to have a distinct heading.

The motion was put to vote and passed.

THE PRESIDENT. I suppose it is understood that when the term "chrome hide powder" is used in this method, it is meant hide powder chromed as per original directions. It does not specify, but it is the sense of the motion that is meant.

MR. REED. That finishes the subject of the analysis of liquors. You come now to Evaporation and Drying, Paragraph 15.

"Evaporation and Temperature." All evaporation and drying shall be conducted in the form of apparatus known as the "Combined Evaporator and Dryer," at a temperature not less than 98 degrees C. The time for evaporation and drying shall be 16 hours.

MR. YOCUM. I move that be adopted.

The motion was seconded.

MR. YOCUM. While personally I have no objections at all to the combined evaporator and dried—I expect to put one in. We have not been using it—We have been using the old water-bath—in which the temperatures are variable from 92 to 96 degrees. I personally have no objections to the evaporator and dried and mean to put one in—intend to do that anyway, but I do not think it ought to become a method which makes the purchase of an apparatus which costs so much as this does obligatory to some members of the Association who possibly are not in a position to do so. I think if there is some way out of this matter that would effect the same result,

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we ought to give those members a chance and not force them to do it at once, first off. This will go into effect immediately upon its adoption here, and it will be impossible for the members of the Association to obtain these driers inside of three or four months and the apparatus would cost from $75 to $100 and I question the equity of dumping this on the Association as such a sudden matter. I would not object at all to making it a provisional method this year with adoption a year from now, but I think it is using the club to some of the members.

MR. REED. The Referee had that in mind when he made his recommendation. He felt and feels very strongly that a great deal of the difference in the past in the returns of the various analysts has been due to not having a universal method of evaporation and drying and it was because he felt so strongly upon this matter and believed so thoroughly in the combined evaporator and drier that he submerged all feeling of the kind Mr. Yocum mentions, for the good of the cause.

I will suggest, if it might be the sense of the meeting,—a resolution might be passed to this effect—that this official method as read and passed upon, go into effect say on January 1st next, or at any date that the convention thinks wise, it would obviate to some extent—

MR. YOCUM. How long does it take to have that apparatus made?

MR. VEITCH. I have not seen the general figures on this question, but it does not seem to me that this combined drier and evaporator should give any different results than the use of the bottom shelf of a large hot-air oven, provided the absolute time of drying after the liquor has been evaporated is the same. I don't see why, for instance, if on the combined evaporator and drier it comes to dryness in two hours and you have to dry a period of fourteen hours in the ordinary bottom shelf of an oven, the result would be the same.

MR. YOCUM. I quite agree with the Referee as to the efficiency of the dryer and the advisability of its use. The remarks made by Mr. Veitch are made, I think, without data. I think the data will hold up the Referee's report, but my objection to the clause read is, that it does not give, and even the first of January would not give, the members of the Association an opportunity to ob-

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tain the apparatus, and if they did have to make a comparative analysis, and if they had not used the method in this particular, and the results were different from those of some other person who had used this, the responsibility would rest on them for not having followed the method. It is hardly fair, and while the improvement of the method always necessitates this and does necessitate it within a year, at the same time it is hardly fair to put the thing in the position that this puts it—that a man who has not this apparatus cannot use the Official Method. I only refer to those who, through lack of time or other reasons, cannot obtain this machine.

MR. SMALL. It seems to me that Mr. Yocum's point is well taken and it seems to me that some provision or amendment should be inserted in this paragraph allowing the members the use of whatever drying apparatus they may have which conforms to the method now used for a matter of six months or something of that sort. At the same time, I do not think the use of the provisions of the method that we have adopted so far, which entail any new apparatus,—we should be deprived of the use of them for six months simply because we are not going to use this particular section of the method in that time. My idea is that this method should go into effect immediately upon its adoption and should be in effect now, but I think Mr. Yocum's point is well taken and some leeway should be given the members for securing the apparatus. It is asking considerable of a man to provide this apparatus at once off-hand.

MR. REED. The Secretary wishes to state that he has been assured that the members can receive this combined evaporator and dryer at cost.

MR. VEITCH. Would it not be well, then, to add this as another method to our present methods of drying—make it optional. We have two methods, I think, now. It is getting it up pretty heavy. We might use four.

MR. YOCUM. I move that amendment be made to this clause to read as follows:

"All methods for drying as adopted in 1904 shall remain in effect until July 1, 1906, when this clause then becomes compulsory."

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And that the drying under the previous method will then go out of use entirely.

MR. SMALL. I second this.

PRESIDENT. In the meantime this has not been made official.

MR. YOCUM. Yes, but in the meantime they will be official until July, 1906. That will give sufficient time for any one.

PRESIDENT. We will now vote on the amended motion.

The motion was put to vote and passed.

MR. REED. "(16) Dishes. The dishes used for evaporation and drying of all residues shall be not less than 2¾ inches diameter, nor greater than 3 inches diameter."

MR. CARR. Does that specify flat-bottom dishes?

MR. REED. No.

MR. CARR. It ought to.

MR. HALEY. Are these dishes copper, platinum or aluminum? Would it not be well to state?

MR. YOCUM. I think it should be specified.

MR. REED. Flat-bottom glass dishes.

MR. VEITCH. As Mr. Small has brought it up, why not specify the glass?

MR. REED. It will not be necessary with the combined evaporator and dryer.

The motion was put to vote and passed.

Upon motion, the meeting adjourned to meet at 2.30 P. M.

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Journal of the

American Leather Chemists Association

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The American Leather Chemists Association

G. A. KERR, W. H. TEAS. . . . . Past Presidents

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VICE-PRESIDENT—C. W. Norris, Milwaukee, Wis.

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REFEREE—F. H. Small, Worcester, Mass.

ASSOCIATE REFEREE—H. T. Wilson, Dubois, Pa.

REFEREE, CHROME SECTION—L. E. Levi, Milwaukee Wis.

COUNCIL—J. H. Yocum, Newark, N. J.; E. J. Haley, New York City; R. P. Cushing, Lock Haven, Pa.;

FOURTH SESSION.

Thursday Afternoon, November 16, 1905.

The meeting was called to order, MR WILSON in the chair.

PRESIDENT *pro tem.* We will now finish the discussion of the Official Method.

MR. REED. "V. Determination of Total Acidity of Liquors.

"(17). Place 100 cc. of the liquor in a 500 cc. flask and make up to the mark with water. To 100 cc. of diluted liquor in a flask with tube condenser add 2 grams of chemically-pure animal charcoal. Heat to boiling temperature with frequent shaking; cook, filter, and titrate an aliquot with decinormal alkali."

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It is exactly the same as it has been. There is no alteration at all.

On motion of Mr. Small it was adopted.

MR. REED. You come now to the method of sampling from bark, unless otherwise agreed between buyer and seller.

A proposed method for sampling was then read by the Refere, followed by prolonged discussion.

MR. ALSOP. I move that this whole subject be reported back to the Committee for further revision. I think we are wasting a good deal of time without arriving at any conclusion.

The motion was passed and Messrs. Small, Teas, Yocum, Haley and Norris were appointed on that committee.

MR. REED. I will have to ask a little instruction on this. Shall we go to work and after we get these revised methods adopt the whole method again?

MR. VEITCH. We have already adopted part of it. I don't think we need go over that again.

MR. VEITCH. I would like to extend an invitation from Dr. Wiley for the members of the Association to visit the Bureau of Chemistry at any time as a body or singly and look around and see what they have down there. There are a number of interesting things—probably not so much along your line of work as other work. But there are a number of interesting things, I am sure, and Dr. Wiley has authorized me to extend the invitation.

MR. REED. I move it be accepted with thanks.

MR. ALSOP. I think this Association should instruct that Committee as to what they want done.

MR. YOCUM. Mr. President, the officers have evidently appointed the Committee with an object in view. Would it not be up to the Council to express to this Society what were the objects for which this Committee was appointed?

MR. REED. One object in appointing the Committee was to alter the present method of voting upon the election of members, which I think is a very important matter. The Secretary has been very much troubled during the past year by the By-Laws, which he drew up himself, in that it required a mail vote. It is a very impracticable way of doing it and I agree with Mr. Alsop. There should be a committee to act upon the applications for membership and I would suggest in that connection, as the By-

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Laws already in force provide for the Council, that it might save trouble by granting them the power of passing upon members.

Another matter that comes within the province of this committee is in connection with the official method of this Association. We have passed now as an official method for the Association upon most of the articles, and there is a clause in our present By-Laws which says that the official method of the A. L. C. A. shall be identical in all respects with the official method of the A. O. A. C. If we adopt this as our official method, that By-Law should become valueless and I believe should be wiped out.

Then, there is the election of officers. It is a little obscure in its present form of By-Laws. It would seem as though some such system as obtains in the American Chemical Society, where a form is sent around at a certain time, previous to a meeting, in which they express their preference for officers and vote upon them if they are not present,—would meet our requirements. That is a matter that the committee should take up and pass upon, and if necessary, change, alter or wipe out entirely any present By-Law that would in any way conflict with the new By-Law. All this, of course, would have to be referred back to the vote of the Association. The committee is appointed to rectify the present By-Laws and report back to this Association, which will pass upon the recommendations.

PRESIDENT *(pro tem)*. The question of a publication of the organization is suggested, and I would like to hear from some of the members regarding it,—a publication to be the representative organ of our Association. Dr. Levi sent on a set of resolutions for the consideration of the Association. They were to the effect that a particular paper be nominated for a year as the official publication of the Association.

MR. ALSOP. The growth of the A. L. A. C., and the interest shown by the members of the collaborative work this year, seems to insure the success of the Association. The admission of Associate members should add materially to the membership, and consequently to the amount of money at its command. It is probable that the collaborative work and the yearly reports would be enough to hold the interest of the Chemists.

I think, however, that we should make the Association as interesting as possible to the Associate and at the same time increase

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the value of the active membership. In my opinion this can best be done by the publication of a Journal, being a record of work done by members and others, articles of interest to the Trade, translations and abstracts of all literature on the subject that can be obtained. This Journal to appear as nearly as possible at regular intervals, say once each month.

Such a publication would be of great value to the Chemist, and also to all interested in the subject. It would also probably be the means of considerably increasing our membership. It could probably be published in one of three ways, directly by the Association, in connection with a Leather Trades Journal, or as a supplement, but published separately, to a Trades Journal.

It would entail, among other things: Subscriptions, by exchange or otherwise, to Journals, etc., having literature bearing on the subject. This in itself would be of value, as the Association would in that way accumulate files of these Journals. A self-sacrificing Editor and two or three Associates who would serve without compensation.

PRESIDENT. In reply to a letter to Dr. Levi I spoke of a publication somewhat similar to that mentioned by Mr. Alsop and to that his reply is as follows:

Letter From Dr. Levi.

MILWAUKEE, WIS., OCTOBER 11, '05.

MR. WM. H. TEAS,
CHEMIST, ELK TANNING CO.,
RIDGWAY, PA.

DEAR SIR:—Your favor of the 9th inst. received. Allow me to thank you for your willingness to present my resolutions to the Association. Regarding the Quarterly would say that my idea on this subject is not very favorable for the following reasons:

1st. A quarterly for the chemist is generally of little value inasmuch as the papers published in the same, with the exception of original articles, are old before the journal (quarterly) is issued.

2nd. The publication of a quarterly is expensive.

3rd. The Association is as yet not large enough to warrant the expense

4th. We want to get the original articles as soon as possible without the least delay.

My reasons for using a trade journal as our official publication are as follows:

1st. Published weekly.

2nd. Large circulation, bringing our Association prominently before the mass of tanners and chemists.

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3rd. Increase of membership of our Association due to bringing the Association weekly before the trade.

4th. No expense to the Association.

5th. Association is young aud must economize, which can be done in this way.

When the Association has a large membership then the time is ripe for a semi-monthly issued by the Association, and then enough advertisements can be obtained to pay all expenses of publication and leave a handsome surplus.

Wishing the Association a most successful meeting and hoping to have the pleasure of meeting you and the gentlemen very soon, I am,

Sincerely yours,

(Signed) LOUIS E. LEVI.

I submit that without any comment. If the Association is willing to consider such a scheme I should think a motion and the appointment of a committee to look into it would be in order.

MR. ALSOP. I move that a committee be appointed to look into the practicability of publishing a journal and the way it could best be done and report as soon as possible to the President, and that the Council have authority to take action upon the report.

THE PRESIDENT appointed Mr. Alsop, Mr. Reed and Mr. Norris as the committee.

Further discussion followed regarding the By-Laws.

MR. SMALL. If it is in order at this time, I would like to read a communication I have had from Dr. Parker, the Honorable Secretary of the International Association of Leather Trades Chemists. Dr. Parker says:

Letter from J. Gordon Parker.

LONDON, S. E., NOV. 3, 1905.

MR. FRITZ H. SMALL:

DEAR MR. SMALL:—

I wish Washington were nearer, as there is nothing I should enjoy so much as to meet the leather trades colleagues in your country. I would like you to tell them if this letter reaches you in time that we are slowly but surely getting less satisfied with out hide-filter method, and that several of us are experimenting on chromed hide-powder, and personally—I can only speak for myself—I have come to the conclusion that chromed hide-powder worked on American lines, or perhaps a modification of those lines, is more reliable than the hide-filter method. I fully expect that at the Prague Conference some method of analysis by means of chromed hide-powder will be adopted. I sincerely hope that your Association may send a delegate over.

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I would ask you to beg your colleagues that they will occasionally contribute something to the "Collegium." Any article dealing with laboratory observations, anything of general interest to the leather trades chemists, however short or however long, will be thankfully accepted and published. We want to draw nearer to one another, and we can do this by interchange of thought through the pages of our journal.

Yours sincerely,

(Signed) J. GORDON PARKER.

I would like to emphasize the two points that are brought out here. One of these is that the men in this Association who are doing work along tannery lines—that is, original—should, so far as possible, send all papers on this subject to the Collegium. It seems to me, as Dr. Parker says here, that a better understanding of the position of the members of the two societies and their differences, and so on, can be brought out in this way. Further, it seems to me that it is distinctly worth while that if possible, some representative of this Association should go to the Prague next year and represent the Association there. Whether it would be possible for the Association to do anything towards defraying the expenses, I do not know. It might be well to consider it here. Where tanning material is being bought abroad and being sold in this country anything that can be done to bring about a common method it seems to me should be done. I would like to call for the sense of the meeting with regard to the possibility of sending a delegate to Prague for the conference of the International Association next year.

PRESIDENT. I think the Secretary should be instructed to acknowledge the receipt of Dr. Parker's letter.

MR. ALSOP. I understand that two of our members are going to attend.

MR. HALEY. I think since Dr. Parker has so kindly suggested that we send to the Collegium some communications concerning the advancements of the tanning interests in this countrty our Honorable Secretary should be instructed to forward to the Honorable Secretary of the International Association a resumé of the comparative determination of tannin by the various analysts of this Association.

DR. CASABURI. I have listened with very much interest to your discussion and I find that this sort of thing is good for us and I agree with Dr. Parker in wishing you would come over to the

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Prague and we will do what we can to make you enjoy your trip and you will get in touch with the different people and get new friends and many difficulties will be smoothed over, and I think we might come to an agreement that would be very well for both Associations. I should like very much to have two or three of you come. I wish you would all come over, but I don't believe it would be possible. You would be perfectly welcome.

MR. J. H. YOCUM then read his article on the "Determination of Nitrogen in Leather."

MR. CLAFLIN. Mr. Chairman, with your permission, I would like to call Mr. Yocum to account. I had a good deal of work on the analysis of leather, partly on account of my connection with the committee. The only question that troubled me was the extraction of tannin. I found that there was no limit to the tannin I could get out. I tried it at 50 degrees C. I think if I had continued the 50 degrees centigrade long enough I might have reached an end, but if you extract until you get a litre there is still a good deal of tannin coming away. If you raise a temperature a little you will get much more, and I found there was a very wide variation of the results of combined tannin. On the nitrogen determination one point came up, and that is, the effect of chlorides. There seems to be an opinion that in a cold bath where chlorides are present there is a loss of nitrogen in leather on account of ammonium chloride distilling off. I think for the average chemist he will get sufficiently accurate methods to use the ferro-cyanide test, instead of determining the copper oxide. It is much simpler. The result in making an experimental determination first gives you fully accurate results.

MR. YOCUM. In reference to our solubles determination I shall only speak of the experience I had in our laboratory. There is no method that I know of that is condensed and accurate. You get yourself in exactly the same position that you do leaching the bark. We take 30 grams and percolate to 1,000 cc. It is not all of it. That is admitted. But you try for another 1,000 cc. and you will still be in the position that you would not have it all off, but you have got to stop somewhere.

MR. CLAFLIN. I think it would be well to have an empirical method.

MR. YOCUM. Well, you could continue that for a long time and

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still have the presence of tannin. You cannot get above 50 degrees C. The leather runs together there.

MR. CLAFLIN. I did not find it so. I tried it with alcohol, my idea being that it might be possible to get a constant residue, a residue that was constant in tannin.

MR. DELANEY. May I ask if you ever tried using methyl alcohol?

MR. CLAFLIN. No.

MR. YOCUM. The trouble is you never get to a point where it is complete. You have to determine your tannin by difference.

I do not know that there is any value to it for all leathers, but occasionally there is. There is a value where you have adulterated leathers.

MR. CLAFLIN. I would try to find out the difference between quick tanned leather and old-fashioned tanned leather. I think it is very important. They are improving the quick tanned leather so it looks like the other leather, and it would be good value for the producer to know which was which.

MR. YOCUM. In reference to the effect of chlorine on nitrogen determination, I have never had any leather that has had chlorine. I have never found it to have any.

PRESIDENT. The Secretary has here an application for Associate Membership. F. C. Klipstein, proposed by E. J. Haley, Mr. Wilson and Mr. Teas. Business importer and manufacturer; address, East Orange, N. J.

Mr. Klipstein was elected.

The motion was then carried to adjourn until the ensuing day at 9.30 A. M.

Fifth Session.

The fifth session was called to order Friday morning, November 17, and the time until noon was devoted to discussion regarding the official method for tannin analysis, and to the position of the Association relative to the A. O. A. C.

The Secretary read the paragraphs he was authorized to add to the Official Method and they were adopted.

On motion, adopted. The Secretary of the Association was added to the Committee on By-Laws.

The meeting then adjourned to attend the meeting of the A. O. A. C.

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Sixth Session.

The sixth convened Friday afternoon, and it was voted that the Association accept the report of the Referee and express their thanks to him for it.

On motion adopted, it was decided to enlarge the field of the Association to include chrome tonnage, and a referee-ship for this branch of the tanning industry was provided for.

Election of officers was next in order and resulted as follows:

Referee: Fritz H. Small, Worcester, Mass.

Assistant Referee: H. T. Wilson, Buena Vista, Va.

Referee for the Chrome Section or Division: Dr. Louis E. Levi, Milwaukee, Wis.

President: H. C. Reed, Stamford, Conn.

Vice-President: C. W. Norris, Milwaukee, Wis.

Secretary and Treasurer: William K. Alsop, New York City.

Executive Committee: John H. Yocum, Newark, N. J.; Robert P. Cushing, Lock Haven, Pa.; E. J. Haley, East Orange, N. J.

On motion adopted, the Secretary was instructed to draw up resolutions expressive of the regret of the Association at the death of Mr. William H. Krug.

After discussion as to the next meeting place, the Convention adjourned *sine die*.

OFFICIAL METHOD OF THE A. L. C. A. FOR TANNIN ANALYSIS.

I. Crude Materials.

(1) *Moisture Determination.*

Upon receipt of the sample, grind promptly and dry 10 grams in the manner and for the period specified for evaporation and drying in extract analysis.

(2) *Preparation of Sample for Extractions.*

Sample must be dried at a temperature not exceeding 60 degrees C., and then ground to such a degree of fineness that the entire sample will pass a sieve of 20 meshes to the inch (linear).

(3) *Amount of Sample and Proportion of Water for Extraction.*

For fresh materials the amount of sample and proportion of water for extraction should be such as to give between .35—.45

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gram tannin per 100 cc. of solution. For Spent Materials this proportion should be approximated as closely as practicable.

(4) *Extraction of Sample.*

Extraction shall be conducted in a form of apparatus that permits the removal of the extractive solution from the influence of sustained high temperature, and shall be continued till a portion tested with gelatine salt solution fails to give a precipitate. At least 400 cc. of the first portions of extractive solution should be removed and not subjected to further heating. Thin layer of cotton must be used in order to prevent fine material passing over.

(5) *Analysis.*

After extraction and dilution, solutions must be heated to 80 degrees C., and analysis conducted as per Official Method for Extracts. In case of weaker dilutions than the Official Method specifies the amount of hide powder must be reduced in proportion to the reduction of tannin.

Ten grams of the air-dried sample should be dried as in (2) to determine moisture content of the portion extracted, and the analysis calculated and reported upon a "dry" basis.

II. Analysis of Extracts.

(6) *Amount and Dilution for Analysis.*

Fluid extracts must be allowed to come to room temperature and weighed in stoppered weighing bottle. Such quantity shall be taken as will give from 0.35—0.45 gram tannin per 100 cc. of solution, dissolve in exactly 900 cc. of distilled water at 80 degrees C., and made up to mark after standing not more than 20 hours, nor less than 12 hours. Temperature must not go below 20 degrees C.

(7) *Total Solids.*

Thoroughly mix solution, pipette 100 cc. into tared dish, evaporate and dry as directed under "Evaporation and Drying."

(8) *Soluble Solids.*

To 2 grams of kaolin (free from soluble salts) add 75 cc. of the tannin solution, let stand 15 minutes, decant as much as possible, add 75 cc. of solution, stir and pour immediately on pleated filter paper No. 590 S. & S., 15 cm. diameter. Keep filter full, reject first 150 cc. of filtrate, evaporate and dry next 100 cc., as per "Evaporation and Drying." The portion for soluble solids determination must be as clear as practicable, and evaporation during

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filtration must be guarded against by covering funnel and receiving vessels for filtrate. The temperature of solution during filtration shall be kept between 20 degrees and 25 degrees C.

(9) *Non-tannins.*

A quantity of hide powder sufficient for the number of analyses to be made shall be prepared in the following manner: Digest with 25 times its weight of water till thoroughly soaked. Add 3 per cent. of chrome alum in solution. Agitate by either shaking or stirring occasionally for several hours and let stand over night. Wash by squeezing through linen, containing the washing until the wash water gives no precipitate with barium chloride. Squeeze the hide, using a press, if necessary, so that the wet hide will contain between 70 and 75 per cent. of water. Use approximately 20 grams of wet hide for moisture determination. Add to 200 cc. of the original solution such quantity of the wet hide as represents from 12 to 13 grams dry hide. Shake for ten minutes in some form of mechanical shaker and squeeze immediately through linen. Add 2 grams kaolin to the filtrate, stir and filter through folded filter (No. 1 F Swedish, recommended) of size sufficient to hold entire filtrate, returning till clear. Evaporate 100 cc. of the filtrate. The weight of the residue must be corrected for the dilution caused by the water contained in the wet hide powder.

The non-tannin filtrate must not give a precipitate with a 1 per cent. gelatine 10 per cent. salt solution.

(10) *Tannin.*

The tannin content is shown by the difference between the soluble solids and the corrected non-tannin.

III. Analysis of Liquors.

(11) *Dilution.*

Liquors must be diluted for analysis so as to give as nearly as possible 0.7 grams solids per 100 cc. of solution.

(12) *Total Solids.*

To be determined as in Extract Analysis.

(13) *Soluble Solids.*

To be determined as in Extract Analysis.

(14) *Non-tannins.*

To be determined by shaking 200 cc. of solution with an amount of wet chromed hide powder, containing 70 per cent. to 75 per

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cent. moisture, corresponding to an amount of dry hide powder shown in the following table:

Tannin range per 100 cc.	Dry Hide Powder per 200 cc.
.35—.45 gram.	8—10 grams.
.25—.35 gram.	5— 8 grams.
.15—.25 gram.	2— 5 grams.
.00—.15 gram.	0— 2 grams.

Solutions to be shaken for non-tannins as in Extract Analysis; 100 cc. must be evaporated as in Extract Analysis.

IV. Evaporation and Drying.

(15) *Evaporation and Temperature.*

All evaporations and dryings shall be conducted in the form of apparatus known as the "Combined Evaporator and Dryer," at a temperature not less than 98 degrees C. The time for evaporation and drying shall be 16 hours. The method of drying adopted in 1904 shall be continued in force till July 1, 1906.

(16) *Dishes.*

The dishes used for evaporation and drying of all residues shall be conducted in flat-bottom glass dishes of not less than 2¾ inches diameter nor greater than 3 inches in diameter.

V. Determination of Total Acidity of Liquors.

(17) *Place 100 cc. of the liquor in a 500 cc. flask and make up to the mark with water.*

To 100 cc. of diluted liquor in a flask with tube condenser, add 2 grams of chemically-pure animal charcoal. Heat to boiling temperature with frequent shaking, cool, filter and titrate an aliquot portion with decinormal alkali.

Experimental Methods.

(A) *Soluble Solids.*

Stir up 75 cc. of the solution with 1 gram of kaolin, pour upon single pleated filter paper No. 590 S. & S., 15 cm. filter and return filtrate for one hour, keeping filter full. Have cooled a sufficient portion of the original solution to 20 degrees C., and after removing solution from the filter proceed with filtration of cooled solution, discarding at least 10 cc. of the filtrate and collecting the first 100 cc. thereafter coming clear.

(B) *Analysis of Liquors.*

Make up solutions for analysis to contain .35—.45 gram tannin

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per 100 cc., dilutions being made with water at 80 degrees C. In each case evaporate such an aliquot portion as will give a residue for soluble solids of approximately from .7—.8 gram.

Determine soluble solids as in Provisional Method A. Determine non-tannins as in the Official Method.

NOTE.—It is understood that these Experimental Methods are not to be used for reporting analyses.

The above method was adopted provisionally by the A. O. A. C. and thus becomes the method for Tannin analysis for the ensuing year.

DETERMINATION OF NITROGEN IN LEATHER.

By J. H. Yocum.

Owing to the fact that I have not had time to give to the work of the Committee on Nitrogen Determination, of which I am Chairman, no results or comparative analyses are presented, but it would seem to me that the question of Nitrogen determination having been gone over so very thoroughly by the A. O. A. C. it leaves little for this Committee to do in this line. It would, therefore, seem to me proper that the A. O. A. C. Kjeldahl method for Nitrogen determination be followed by this society.

The method of the A. O. A. C. will be found in the reports of the proceedings of their various conventions from 1894 to the present date. This particular method of Nitrogen determination is available for use to all chemists and is a method which permits of no discussion as to its accuracy and availability, and it would seem to me that this Society should adopt as their own the method adopted and approved by the A. O. A. C.

The determination of the percentage of nitrogen in a sample of leather is only a part of what I believe to be the proper domain of this Committee, it being quite as much a part of this Committee's work to determine the nitrogen in pure hide as in the leather itself, for without the determination of the nitrogen in pure hide no relationship between Hide and Leather can be established.

Messrs. Von Schroeder & Paessler in the Dingley Polytechnic Journal in the year 1893 made extensive determinations of the nitrogen in pure hide substance from various sources. So far as relates to the analyses of leathers which would normally come

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to the chemist we find from their determinations that pure Calf, Ox, Horse, etc., is 17.8 per cent., while that of Goat is 17.4 per cent., and Sheep 17 per cent., these representing practically all the Hide sources of commercial leather, and as their work is generally accepted and used extensively, it does not appear to me that it is necessary for us, at the present time at least, to go further than to also accept their results. It might be wise at some future period to corroborate this work, but I find that these figures are accepted universally abroad and in this country, that the possible error is very small, and it is hardly worth the amount of work which would be necessary to re-determine them under present conditions, and I would, therefore, suggest that this organization adopt these figures as the figures to be used in determining the amount of hide substance in leather.

I have had my attention called to the preparation of leather samples, by other members of the Committee, some suggesting the determination in the raw leather, others suggesting preparatory treatment of the leather before the Nitrogen determination.

It would appear that in ordinary sole leather where the percentage of nitrogen containing fats are low, the Nitrogen determination would not be materially influenced by the Nitrogen contained in the fats held by the leather, but that on the other hand in upper leathers which contain large proportions of grease, and belting and harness leather which are stuffed heavily with grease and in Goat skins which contain large proportions of oil, a preliminary removal of the fats would make certain the preliminary removal of fats from the leather.

I therefore suggest that the following procedure be followed. That a considerable sample, 8 to 16 oz. of leather be thoroughly broken down either by cutting or grinding into as fine a condition as possible, that a fair sample of this broken down leather be drawn of say, 40 grams, and that this be extracted with petroleum ether, the fats and oil being determined from the ether residue. That this leather be then thoroughly dried and a sample of it used for the Nitrogen determination. This, to my mind, will make a uniform rule for leathers of all character, whether they be full of grease or be comparatively free from grease.

I believe that the proper scope of the Committee's work should not be covered by the term, "Committee for Nitrogen Determina-

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tion in Leather," but by the term, "Committee on the Analyses of Leather," as the Nitrogen determination is only a part of what is represented by leather analyses. Actually the object of making a Nitrogen determination is to determine the amount of hide substance in a given sample of leather, and consequently the ultimate aim is not to determine nitrogen in leather, but determine hide substance, and also if a request for a hide substance determination is made it means to carry the other contents of the leather.

In addition to this there is another condition which exists which must be considered, and that is that a portion of the tannin in leather is combined with the hide and that there is another portion which is not combined with the hide and which is removable by digestion with water, and with this removable portion will always be found a certain amount of non-tannins, so that we really have beside the oil, ash, moisture and hide substance determinations, determination of adulterants (Grape-Sugar, Glucose, Epsom Salt, Barium, etc.), combined tannin, uncombined tannin and non-tannins. These determinations applying to leathers are made from organic tanning materials. In the analysis of mineral tanned leathers, however, it is usually not necessary to make a hide substance determination for the reason that the determination of the oil, moisture and mineral tanning materials represent all the substance other than hide, which go to make up the leather.

PREPARATION OF SAMPLE.

The sample of leather should be first brought into a state of fineness, either by grinding in a mill or shaving with a knife. Sufficient leather should be brought into this sub-divided condition to obtain therefrom a representative sample. Leathers which contain grease cannot be ground satisfactorily, but if the grease is extracted first in a part of the leather which has been cut into small pieces with a knife it is then easily ground.

For obtaining a sample to make the fixed tannin, uncombined tannin, non-tannin and nitrogen (hide substance) determination, it is best to extract the grease in this manner and grind the sample in a grinding mill.

For the moisture, oil and ash determination and determination of adulterants alone, such as grape sugar, glucose, Epsom salt, barium, etc., it is necessary to draw a sample from the pieces which have been broken down by the aid of the knife.

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DETERMINATION OF MOISTURE.

The determination of moisture is best made by taking a cut-up sample, which is a representative of the leather, and drying at 100° C. It must be borne in mind, however, that most leather has oils or grease of some kind in it, which, under a high temperature, oxidize. Where great accuracy is desired, the drying should take place in vacuum. Usually this is not necessary, as the determination of moisture by drying at 100° C. is satisfactory in all commercial analyses.

THE DETERMINATION OF OILS AND FATS.

In the determination of oils or fats, 40 grams representative of the whole sample is as finely divided as possible by means of the knife. This should be placed in a Soxhlet extractor and extracted with petroleum ether. The solvent remaining in the bottom of the Soxhlet is then filtered, the filter carefully washed with petroleum ether, the petroleum ether evaporated, leaving residual oil or grease. This method of determination is perfectly satisfactory for commercial purposes, but where absolute accuracy is desired, evaporation of the petroleum ether should take place under a vacuum, and consideration must be taken of the character of the oil removed from the leather and the possibilities of it having formed substance insoluble in petroleum ether through oxidation.

DETERMINATION OF WATER SOLUBLE MATTER.

The residue of the 40 grams of leather which have been extracted with the petroleum ether, as above described, is then ground, and from this thoroughly ground mass a sample of 30 grams is taken and extracted in a Proctor's extractor with distilled water at about 50° C. until a liter of the washings is obtained. Regular tannin determination is then carried on this solution, from which is determined the uncombined tannin and non-tannins.

NITROGEN DETERMINATION.

The determination of nitrogen should be determined in a portion of the oil-free leather obtained by extracting the original leather in the Soxhlet with petroleum ether and which should be thoroughly ground and should be from the same source as the sample used for water solubles. If the water soluble matter is

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excessively high, it is necessary to make a determination of the sugars which may be termed adulterants.

SUGAR DETERMINATION.

Usually, in sole leather at least, leathers which contain 5% water soluble matter have the sugar determination run on them as a check, natural sugars in leather rarely exceeding ½ of 1%.

Part of the 1000 cc. obtained from extracting of the fat free leather is precipitated with a good excess of basic lead acetate, filtered, and the lead precipitated with sulphuric acid and filtered. The filtrate is neutralized and made up to a given volume and determination of the sugar made by Fehling's solution and determined according to Ahlin's tables from the weight of precipitated copper.

ASH DETERMINATION.

The ash in the original sample of cut-up leather is determined by incineration and weighing of the ash. Usually from 10 to 20 grams are used. Where the ash exceeds .30% of the weight of the air-dry leather this amount is sufficient to justify further examination. This examination generally is to determine magnesium sulphate, sodium sulphate and barium chloride and a complete analysis is necessary where the ash is excessive.

The ash content admit of interpretation as to whether magnesium sulphate, sodium sulphate, barium chloride (barites, Epsom salts, etc.), have been used for adulterants. It also indicates whether potassium, calcium and sodium sulphate are in excess and in conclusion what percentage of sulphuric acid has been used as a plumping agent.

The determination of fixed tannin is made by difference between the sum of the percentages of moisture, ash, fat, total water solubles and hide substance and 100%, figuring the ash in case of adulterated leather to the theoretical amount of Epsom salt, Glauber salt or barium, as indicated by the complete ash analysis.

Aside from the mere matter of determining adulterations in leather, the complete analysis of it is a good indication of the efficiency of the methods used in its production, and in a general way indicates the gain made in the leather which is of much importance to the tanner who wishes to compare tannages.

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CHROMIUM AND ACID IN SINGLE BATH LIQUORS.

By F. W. Alden.

The percentage of chromium together with the acid combined with the same are the principal things that a chemist needs to determine in analyzing single bath chrome liquors.

If the percentage of chrome is unknown it is evident that the tanner will have very little idea of how much of the liquor to use, and if the proportion of acid is unknown he will find it almost equally difficult to adjust himself to the conditions.

Accuracy is, of course, very important in making an analysis, but it sometimes happens that speed is of such great importance that accuracy can be sacrificed to some extent. In such cases we would recommend the following method for the estimation of chromium as being the most rapid one we know of and, at the same time, sufficiently accurate for practical purposes.

Weigh or pipette off such an amount as will contain between two and three grams of Cr_2O_3; dilute to 500 cc. with distilled water, and after thoroughly mixing, draw off 10 cc. into an Erlenmeyer flask of about 200 cc. capacity. Add a little water (about 15 cc.) and drop in about two grams of sodium peroxide, mixing the contents immediately. The chromium is oxidized almost instantly by this treatment, but a second, and even a third portion of peroxide may be added to make sure that the reaction is complete. The sides of the flask are now washed down and the contents boiled for a few minutes in order to decompose all the excess of peroxide. A small funnel should be placed in the mouth of the flask to prevent loss from the spray flying out of the flask. When this is done, cool the contents under a tap and acidify with strong hydrochloric acid, adding it slowly until the change of color from the light to the dark yellow indicates the formation of the acid chromate. Then add about 4 cc. more of the acid, and after again cooling it is ready to be titrated with $N/_{10}$ sodium—thio-sulphate in the presence of potassium iodide in the usual way.

When greater accuracy is required, we prefer to precipitate with a slight excess of ammonia, boil, filter, dry the precipitate and oxidize to the chromate in an iron crucible with sodium peroxide, finally adding water, boiling to decompose the excess of peroxide and titrating with $N/_{10}$ of sodium-thio-sulphate as before.

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It has been our experience that to ignite and weigh the precipitate hydrate of chromium invariably gives too high a result owing to the difficulty of thoroughly washing the precipitate unless it is re-dissolved and precipitated two or three times.

The determination of chromium is fairly easy, but the real difficulty lies in the determination of the acid combined with the chromium. The following method is the only one we know of which estimates the acidity of chromium salts directly. As it is based on the precipitation of chromium hydrate with a known quantity of alkali it is not applicable to such salts of chromium as are not precipitated in this way. The difficulty arises from the fact that when chromium is precipitated with an excess of alkali, it carries with it a part of the alkali either mechanically, or feebly combined so that as the alkali for precipitating the chrome is increased the amount apparently neutralized is also increased. And, on the other hand, the chromium may be entirely precipitated by less alkali than is required to combine with the acid owing to insoluble basic salts being thrown down.

After a number of experiments it was found that an excess of alkali within a certain limit would give working results. In order to determine the correct proportion of alkali to be used a preliminary experiment is made with a large excess. Of course, the result obtained is somewhat too high for the reason already explained, but for the second and final experiment an amount of alkali is taken as will just neutralize the acid which is apparently found by the first experiment. It is assumed that this second excess is within the required limit and that the result obtained is correct. After trying various alkalies it was found that sodium carbonate was the best precipitant. The method is carried out in the following manner:

Fifty cc. of $N/_2$ sodium carbonate are diluted to about 150 cc. with distilled water and brought to a boil. Fifty cc. of the diluted chrome liquor already mentioned are then slowly run from a pipette, the sodium carbonate solution being constantly stirred in the meantime. The mixture is again brought to a boil and then cooled and diluted to 500 cc. Two hundred cc. are then filtered off and the alkali titrated back with N_2HCl. This result multiplied by $5 - 2$ and subtracted from the 50 cc. alkali originally taken shows the amount of alkali that was apparently neutralized

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in precipitating the chrome. A second determination is now made, using this amount of alkali and proceeded with in exactly the same manner as the first. From the result then obtained the percentage of acid is readily calculated.

AN EXTRACTOR FOR TANNING MATERIALS.

By H. C. Reed.

Upon careful consideration of last year's report relative to extraction, the writer of this article decided that the best solution of the problem might result from the use of an extractor designed to answer all the purposes required, as to temperature, removal of extracted matter, etc.

With this in view, he designed, had built and has operated, the apparatus which he now presents for your consideration. The description of this apparatus is as follows:—

In the accompanying drawings, Fig. 1, is a vertical section of my improved apparatus showing its various features of construction, and Fig. 2 is a reduced detail of a modified form of cup. In the drawings, A represents a cylindrical enclosing case or vessel having a solid bottom 2, and provided at its top with a flange 3, upon which is seated a corresponding flange 4 of a conical or tapering head 5, the flanges being united by means of bolts 6, nuts 7 and thumb screws 31. A suitable condensing apparatus, not shown, is arranged in the central opening 8 of the head 5, and permanently attached thereto. A pipe 9 is arranged at the bottom of the vessel A, having a suitable cock 10, and connection with a vertical pipe 11, having a graduated gauge glass 12, a funnel 13 and stop-cock 14.

The vessel is also provided with a pipe connection 15 at about one-quarter or one-third of its height from the bottom, having a stop-cock 16 and a faucet 17, connected with which is the vertical pipe 18, the top 19 of which is bent at right angles with it and enters the vessel near the top. An overflow faucet 20 is arranged in the vessel above and to one side of the pipes 18 and 19.

Arranged about the middle of the vessel A is the inner circumferential flange 21, upon which is removably arranged a reticulated partition 22. Upon this partition is removable seated the cup

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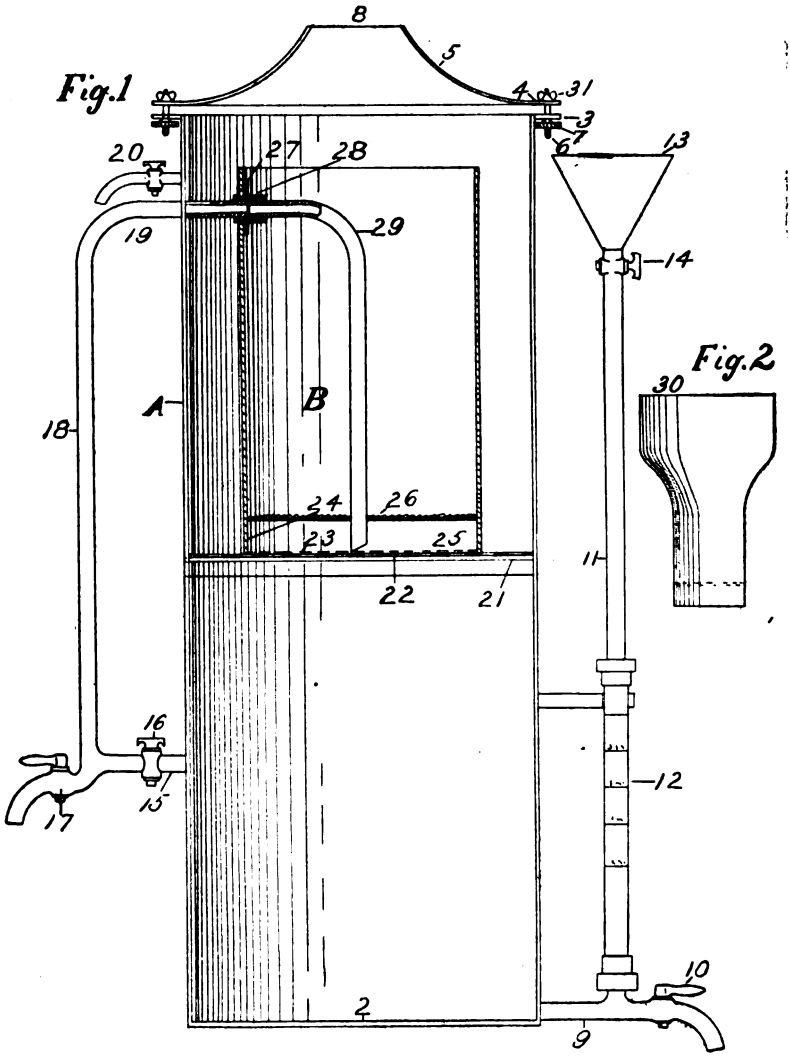
B having a closed head or bottom 23. Twenty-four is a cylindrical sheet-metal ring fitted in the bottom of the cup and having per-



manently secured thereto perforated circular plate 25 which supports a fine wire mesh sheet 26. The end of the pipe 19 extends through an opening in the side wall of the cup B wherein it is

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rigidly and tightly secured by means of an exterior gasket 27, and an interior collar nut 28 threaded thereon. The projecting inner end of the said nut is flared conically to receive the tapering conical end of the bent pipe 29, the lower end of which extends to the bottom of the cup B, its tip being cut on a bevel so as to afford free inlet to the tube. The two tubes so united constitute a siphon for the draining of the cup.

When it is desired to use the apparatus for treating small quantities of material for which the ordinary cup B would be too large, I substitute for it the cup shown in Fig. 2 in which the top portion 30 is cylindrical and of the same size as that of the cup B and provided with a similar opening for the receiving pipe 19, but the side walls are contracted as shown, so that the lower end of the cup is of much smaller diameter.

The method of using the apparatus is as follows—the parts being assembled as shown by the drawing:

The bark or other material, suitably prepared for treatment, is introduced into the cup B and rests upon the wire mesh screen 26 and prevented by it from passing to the bottom of the cup. If it is desired to extract the same at a temperature less than that of boiling water, water at the desired temperature is introduced into the vessel A through the funnel 13 and pipes 11 and 9, or cold water may be thus introduced to the vessel and the temperature thereafter raised by a heater placed under the bottom of the vessel A. The water is then run into the vessel until it overflows through the faucet 20, thus surrounding the cup B with water at any desired temperature not exceeding 212 degrees Fahrenheit. Water can be induced into the cup B by pouring it into the same or by closing the overflow faucet 20 and allowing the water to rise in the vessel until it shall overflow the edge of the cup B and fill the same, or by means of an inverted vessel filled with water, the open mouth of which dips below the level of the water in cup B and which automatically replenished the water in cup B at a rate equivalent to the lowering of the level of the water in said cup. The water percolates through the material in the cup carrying the soluble matters therefrom through the screen to the bottom of the cup. When the water in the cup rises above the top of the siphon, the solution in the bottom of the cup will flow through the siphon and may be drawn off through the faucet 17.

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The rapidity of percolation and outward flow through the siphon is controlled at the faucet 17.

If it is now desired to extract by means of condensed steam the faucet 10 is opened and the water in the vessel is drawn down to the desired level indicated by the gauge glass 12. The faucet 10 is then closed and the condenser attached to the top of the vessel A. Heat is applied to the bottom of the vessel A and the steam from the water therein passes through the partition 22 and around the cup B into the condenser, whence the condensed water drops back into the cup B upon the contained material. The cup B is thus gradually filled by the water until the siphon is covered, when the liquor is siphoned out of the bottom of the cup in the manner described. If continuous extraction is desired, the faucet 17 is kept closed and the cock 16 opened to the desired degree, thus maintaining circulation through the apparatus.

It has been found advantageous to vary the size of the cup with the bulk of the material to be extracted, and for this reason cups of the style shown in Fig. 2 were designed. When small amounts of material are extracted a greater mechanical action results from the use of a cup of a size only sufficiently large to comfortably contain it, and better extraction results.

These points are claimed for the apparatus:—

1. That with it extraction can be made at ordinary temperature (or lower if desired) up to steam heat.

2. That extraction can be made with condensed steam and the extractive removed in any amount desired—thus doing away with the harmful effects produced by continued boiling.

3. That extraction can be made by continuous operation as with the Soxhlet or similar apparatus.

4. That the completeness of extraction can be determined at any stage of the operation.

5. That whether the apparatus is in proper working order may be ascertained almost to a certainty at any time during extraction.

6. That in case of manipulation it is superior to other forms of extractors when the range of temperature within which it will extract, is considered.

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7. That the construction permits of the disconnecting, cleaning and removal of the various parts.

In order to demonstrate the value of the apparatus by actual extraction, identical materials were extracted with it and the copper Soxhlet and the results are tabulated below.

An examination of the table discloses a few interesting facts relative to extraction. For example,—if we should concede to the Soxhlet extractions of the two Mangrove barks, the Mangrove bark, Mallet bark, Quebracho wood, Chestnut wood (2), Hemlock bark and Wattle bark,—the soluble solids values given by the extractions from the new extractor, and from these values subtract the respective non-tannin values of the Soxhlet extractions, we would obtain a tannin value in each instance lower than shown by the extraction with the new apparatus; or, in other words, without granting any increase in non-tannins from the Soxhlet extractions, corresponding to an increase in soluble solids, it would be plainly impossible allowing that maximum extraction had not been attained, to obtain the same tannin value from the Soxhlet as from the new extractor, in the instances specified, when ultimate extraction was accomplished. If in the extraction of the Chestnut wood (1) and the two samples of spent Quebracho wood, we allow an increase in non-tannins agreeing with an increase in soluble solids, when granting the soluble solids of the extractions from the new extractor to the Soxhlet extractions, keeping the non-tannin values of the latter intact, as before, then, in these three instances, the results from the Soxhlet will be found inferior. In the case of the Oak bark only can the Soxhlet extraction be possibly figured as superior.

Attention is directed to a point that would appear well worthy of consideration when extractions are so conducted as to remove the first fractions of the extractive from the influence of sustained temperature. This is in respect to the throwing out of solution of a relatively larger percentage of insolubles owing to the higher concentration of the earlier fractions. It is very plainly evinced in the case of the Quebracho wood extraction, shown in the table. Warming of the solution to 80 degrees C., after extraction and assemblage of fractional extractions, would presumably effect the result desired in the majority of cases, but very probably not in all. Sumac, for instance, would be likely to

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show a loss in tannin from such procedure. The point in question was not considered in conducting the analyses shown in the table.

The influence of the preparation of material upon the resulting extraction is sufficiently exploited in the referee report this year. The personal view of the writer is that no general method will even suffice for the extraction of tanning materials. Given as versatile an extractor as the one just described, presenting the opportunities it does for extraction under widely varying temperature conditions, we are confronted with the question as to the choice of several methods of manipulation. Shall we begin our extraction by percolation at laboratory temperature, gradually raise to a boil and finish by means of steam condensation; shall we rather immediately proceed with extraction by steam condensation, removing a portion of the extractives and finishing by continuous operation; or would it perhaps be preferable to attempt another of the several methods of procedure open to us? These are questions that experimental research alone will answer, but it seems safe to assume that the variety of materials (and it is a constantly increasing variety) will require a variety of methods, and the only solution of the problem would seem to lie in ascertaining and specifying for each the system to be pursued.

TEAS PERCOLATOR EXTRACTOR.

By W. H. Teas.

This form of extractor is designed to operate similarly to the conditions existing in leaching on the commercial scale. It is simply a cylinder B surrounded by water encased in the jacket A. A reservoir located above the extractor supplies the water for the extraction, and also serves by means of the pressure exerted on the water in the jacket, to raise the boiling point of the water in the jacket.

The perforated disc C on the end of the stiff brass wire D serves as the false bottom of the miniature leach; when extraction is completed the extractor is emptied by lifting out the disc and with it the column of bark or wood.

Water dripping through the cock F goes through the column of material and is delivered through the spout E. The position of

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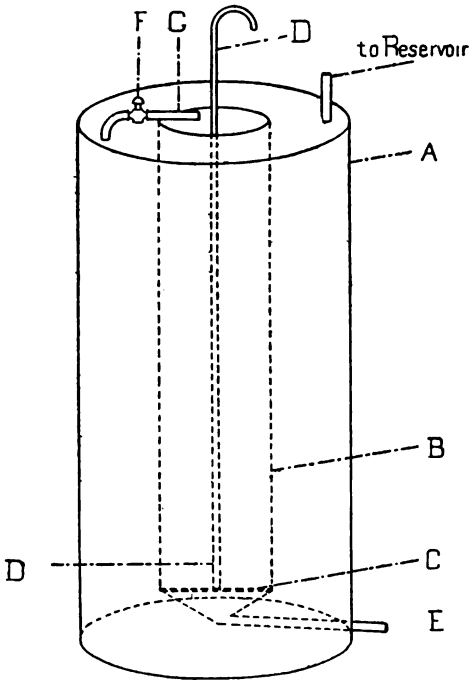
the end of a rubber tube on the spout regulates the height of the liquor in the cylinder B, and the material may be flooded to any desired height by simply raising or lowering the delivery end of the rubber tube attached to the spout E.



Teas Percolator Extractor.

To operate, the nipple G is removed, the wire D withdrawn, the disc C covered by a piece of piano felt, and then replaced in the cylinder, and the material to be extracted is either washed in or run in dry. The jacket and reservoir being filled, the nipple G is replaced and heat applied to the bottom of the extractor. When the desired temperature is reached, the cock F is opened and the proper amount of water allowed to drip on the material. If a true percolation is desired, a layer of cotton or of filter paper is placed on the top of the column of material and the extractives are collected direct from the spout E. If a flooded percolation is desired, the use of the cotton or filter paper may be omitted, but the rubber tube is attached to the spout and the height of the

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liquor in the cylinder B is regulated by the height at which the extractives are delivered.

In the case of spent materials extraction at boiling temperature is maintained from the start. With new materials the extraction is started at the particular temperature suited to the material, and gradually raised to boiling.

A short experience will suffice to enable the operator to judge as to the rapidity of the drip, to insure the required volume of extractives in a given period; and the same may be said as to the regulation of the heating flame after the maximum temperature has been reached.

The height and capacity of the reservoir may be varied to suit local conditions, but the reservoir should be at least of sufficient size to hold a gallon more than the requirements for extraction, and should be at a height that will allow of the water in the jacket being raised to 214° F.

The extractor illustrated is a single unit; circular groups of as high as ten cylinders enclosed by a common jacket fed from a common reservoir, can be conveniently and economically operated.

A Modified Form of the Preceding Extractor.

Since designing the extractor just presented for your consideration, it occurred to the inventor that a modification and simplification in the construction of the apparatus might be beneficial, more particularly where it was desirable to extract by percolation. With this in mind he has developed an extractor to the description of which he now asks your attention.

In the accompanying drawing the outer and enclosing vessel "A" and arrangement for filling same, are in all respects identical with the apparatus first described. The inner cup "B" is furnished with a heavy bottom, permitting a threaded inlet "7." The tube "3," bent at "20" as shown in the drawing, is similarly threaded at "1," and passes through a perforation in the supporting plate "8." By screwing down cup "B" on tube "3" a tight joint is obtained.

Tube "3" passes through wall of containing vessel "A" at "19," at which point it is irremovably attached to "A," and bending

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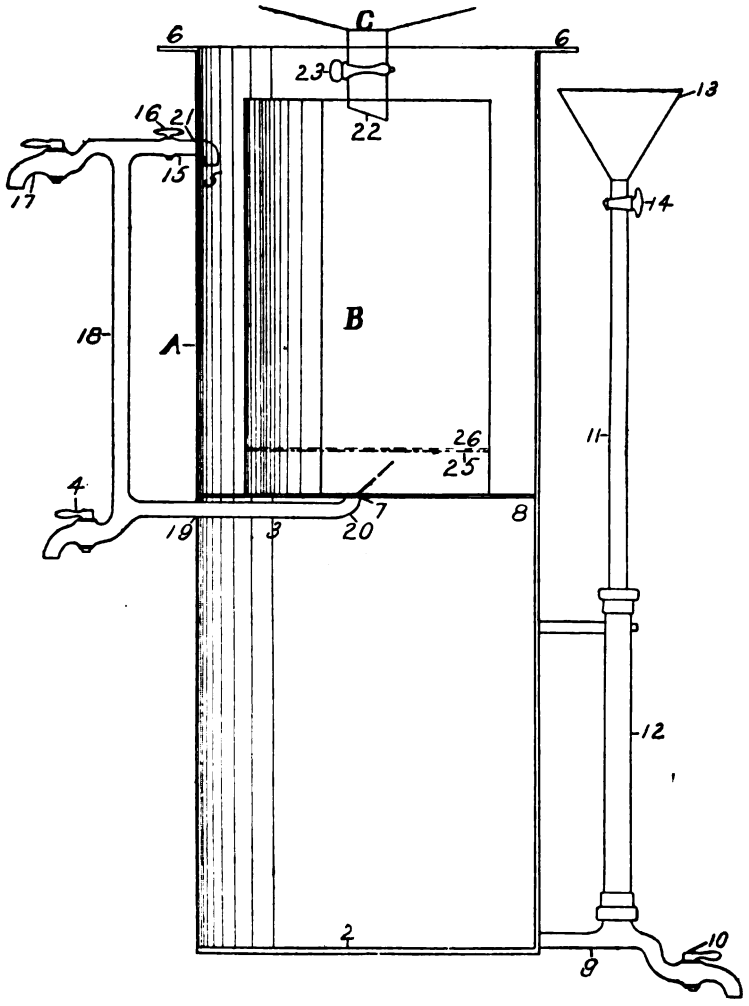
rises perpendicularly, and again bending, re-enters vessel "A" at "21" and is firmly and irremovably attached to "A" at this point of entry.



Faucets "4" and "17" and cock "16" are located as shown in the drawing.

The inner cup "B" has a fine wire mesh screen "26" as in cups used for like purposes, but the screen is not perforated to admit

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tube, differing in this respect from preceding extractor, being without siphon tube.

A condenser may be attached at "6" as in other continuous extractors.

METHOD OF MANIPULATION.

For percolating at any temperature up to steam heat, the finely ground material is introduced into cup "B," resting upon and held by screen "26." An inverted vessel "C," filled with water, is suspended over cup "B," with single outlet "22" of said vessel within cup "B" and at a higher level than that of tube "15." Cock "23" of vessel "C" is opened and water allowed to fill cup "B," covering material therein contained, the flow of water ceasing automatically when mouth "2" of vessel "C" is closed by rise of water in "B." Water is brought into vessel "A," surrounded cup "B," and kept at the desired temperature as hereinbefore described. Cocks "16" and "17" being closed percolation is conducted through faucet "4" at any desired rate, the water in cup "B" being kept at a constant level and automatically replenished by vessel "C."

If it is desired to carry out the extraction by steam condensation, vessel "C" is removed, condenser attached at "6," and water in "A" drawn down by means of faucet "10" to the desired level indicated by graduated gauge glass "12." Cocks "4" and "16" being closed and "17" opened, the required portion of extractive may be collected outside extractor and not subjected to further temperature.

If continuous operation is desired both cocks "4" and "17" are closed and "16" opened, whereby the extractive is returned to lower part of vessel "A."

You will remark the absence of a siphon tube in this form of apparatus. The system of working is to an extent similar to that of the so-called "press" system of leaching. The material extracting is perforce at all times covered with the extracting medium, which would seem a desirable feature. The extractor, similar to the one previously described, permits of the removal and testing of solution to ascertain the degree of extraction, and presents the same means of determining whether the apparatus is working rightly.

Discussion is invited as to the relative merits of the two forms described.

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SIPHON SOXHLET COPPER EXTRACT VS. REED EXTRACTOR.

	Total Solids.	Soluble Solids.	Insolubles.	Non-Tannins.	Tannins.	Amount Extracted. Gms.	Time. Hours.	Preparation as Mesh.	Extractor.	Method of Extraction.
Mangrove Bark (1)	59.17	58.61	.56	9.05	49.56	9	10	8	Soxhlet.	Continuous extraction.
	60.58	59.66	.92	8.85	50.81	9	10	8	Reed.	Steam condensation 500 cc. outside, then continuous.
Mangrove Bark (2)	38.31	35.15	3.16	11.64	23.51	15	10	12	Soxhlet.	Continuous extraction.
	39.83	35.98	3.85	11.42	24.56	15	10	12	Reed.	Steam condensation 500 cc. outside, then continuous.
Mangle Bark	49.84	44.51	5.33	16.11	28.40	15	15	40	Soxhlet.	Continuous extraction.
	51.19	44.41	6.78	15.55	28.86	15	15	40	Reed.	Steam condensation 500 cc. outside, then continuous.
Oak Bark	15.97	14.72	1.25	6.15	8.57	40	10	8	Soxhlet.	Continuous extraction.
	17.88	16.51	1.37	7.11	9.40	40	10	8	Reed.	Steam condensation 500 cc. outside, then continuous.
Mallet Bark	58.01	55.77	2.24	14.12	41.66	9	10	12	Soxhlet	Continuous extraction. (Extraction not complete in 7 hours.)
	57.43	56.39	1.04	12.53	43.86	9	7	12	Reed.	Percolation (150 cc.—75 deg.—80 deg.); (250 cc.—80 deg.—100 deg.), then continuous.
Quebracho Wood	23.00	22.22	.78	2.74	19.48	18	10	16	Soxhlet.	Continuous extraction.
	24.69	22.61	2.08	2.28	20.33	18	10	16	Reed.	Steam condensation 500 cc. outside, then continuous.
Chestnut Wood (1) . . .	9.57	8.85	.72	4.25	4.60	50	10	8	Soxhlet.	Continuous extraction.
	11.10	10.62	.48	4.68	5.94	50	10	8	Reed.	Percolation (250 cc.—75 deg.—80 deg.); (250 cc.—80 deg.—100 deg.), then continuous.
Chestnut Wood (2)	14.17	12.83	1.34	6.68	6.15	40	19	20	Soxhlet.	Continuous extraction.
	14.65	13.30	1.35	6.36	6.94	40	19	20	Reed.	Cold percolation 200 cc.; outside condensation 300 cc.; then continuous.
	15.47	13.91	1.56	6.69	7.22	40	19	40	Reed.	As above.
Hemlock Bark	22.40	19.21	3.19	7.71	11.50	25	15	20	Soxhlet.	Continuous extraction.
	22.91	19.90	3.01	7.68	12.22	25	15	20	Reed.	Steam condensation 500 cc. outside, then continuous.
Wattle Bark	40.65	39.81	.84	11.51	28.30	12	8	12	Soxhlet.	Continuous extraction.
	41.81	41.02	.79	10.54	30.48	12	2¾	12	Reed.	Condensation 1,000 cc. outside only.
Spent Quebracho Wood (1)	. . .	4.29	. .	2.70	1.59	50	15	8	Soxhlet.	Continuous extraction.
	. . .	4.59	. .	2.86	1.73	50	15	8	Reed.	Steam condensation 500 cc. outside, then continuous.
Spent Quebracho Wood (1)	. . .	3.60	. .	2.11	1.49	50	15	40	Soxhlet.	Continuous extraction.
	. . .	4.21	. .	2.18	2.03	50	15	40	Reed.	Steam condensation 500 cc. outside, then continuous.

(1), (2)—Not identical samples.

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Journal of the

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Vol. I. APRIL, 1906 No. 4

W. H. TEAS, ... Editor
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A. W. HOPPENSTEDT, } . . . . . Associates
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THE ANALYSIS OF COMMERCIAL LACTIC ACID.

By Prof. Dr. M. Philip.

Report of the German Section of the I. A. L. T. C. of December 3, 1905. Translated from The Collegium, No. 198, Page 88.

Since lactic acid has gained a permanent place in the leather industry, as a deliming and plumping material, the valuation of the same deserves greater attention.

The analysis of lactic acid must be carried out in two directions:

1. The determination of the percentage, and
2. The determination of the purity.

The percentage of lactic acid can not be determined by simple titration, as in the commercial product besides free lactic acid; there is always present a larger or smaller amount of lactic anhydride. This lactic anhydride is formed from lactic acid even on standing in the cold, according to the formula

$$\begin{array}{l} \qquad\qquad\qquad\qquad\qquad\quad CH_3 \\ \qquad\qquad\qquad\qquad\qquad\quad | \\ CH_3.CH(OH)COOH + (OH)CH - COOH = \\ \qquad\qquad\qquad\qquad\qquad\quad CH_3 \\ \qquad\qquad\qquad\qquad\qquad\quad | \\ \qquad\qquad CH_3.CH(OH)COOCH - COOH + H_2O \end{array}$$

from two molecules of lactic acid, one molecule of water separates. Lactic anhydride is a one valent acid and needs, (as 1 molecule of anhydride is equal to 2 molecules of lactic acid), only one-half as much alkali for neutralization as the acid does. Consequently, if the direct titration with normal alkali would be taken as the percentage of the commercial product, then the anhydride would only be determined as lactic acid, and as it is used in very dilute solution as a deliming material, it seems to gradually split

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up into lactic acid again, and thereby forms from one molecule or one alkali equivalent two molecules or two alkali equivalents. Consequently, only one-half of the value of the lactic anhydride would be determined.

This just mentioned breaking up of the anhydride molecule in two molecules of lactic acid is apparent from the start, on account of the well-known property of this anhydride to form only very unstable salts, and when in contact with alkali, immediately to go over into lactic acid. This is further proven by an observation recently published by Weiss[1], although he lacked the explanation. On allowing the Wislicenus aluminum oxide to stand with very dilute acids, Weiss used, with sulphuric acid and acetic acid, before and after the treatment, the same amount of N/10 alkali for neutralization, while lactic acid, after standing with aluminum oxide, apparently on account of the decomposition of the anhydride to lactic hydrate, needed considerably more alkali than before.

It consequently seems justified, also in practice, to determine the value of commercial lactic acid according to the total percentage of acid, as it presents itself after the breaking up of the anhydride.

Accordingly the analysis of lactic acid should then be conducted as follows:

Fifty gms. or 50 cc. are diluted to 500 cc. with distilled water and 50 cc. of this solution titrated with normal alkali and phenolphthalein to a red color. A measured excess of normal alkali is then added, the whole boiled a few seconds, and then titrated back again with normal sulphuric acid until the red color disappears. The total amount of normal alkali used, less the amount of normal sulphuric acid used for titrating back, multiplied by 1.8 gives the total amount of lactic acid, while from the amount of normal alkali first used, the amount of free lactic acid is calculated in the same way in percentages by weight or volume or gms. in 100 cc.

Although these figures correspond to the practical effect of lactic acid when used in the tannery, they do not correspond to its real composition, as under "free lactic acid" is included also one-half of the lactic anhydride which acts as a one valent acid, although equivalent to 2 molecules of lactic acid. If it is desired

[1] Gerber 1905, page 261.

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to express the percentage of commercial lactic acid in lactic hydrate and lactic anhydride, the amount of alkali used for splitting up the anhydride, which is the difference of the two titrations, must be multiplied by 3.42 to obtain the percentage of anhydride, while the amount of normal alkali used for the first titration, less the amount of the anhydride as a one valent acid, multiplied by 1.8 gives the real percentage of lactic hydrate.

But these values are only correct if the acid present is really lactic acid, and not mineral acid or some other organic acid. As lactic acid is manufactured principally by decomposing calcium lactate with sulphuric acid, a preliminary test for free sulphuric acid should first be made.

O. Eberhard gives for this the following method: One part of lactic acid is shaken in a test tube with 5 parts of 95% alcohol. After allowing the same to stand for a quarter of an hour, 5-10 cc. are filtered off and to the clear filtrate are added a few drops of a 10% solution of calcium chloride acidulated with hydrochloric acid. A quickly appearing turbidity indicates free sulphuric acid.

For the quantitative determination of the free sulphuric acid, it is necessary to—

1. Determine the total sulphuric acid, and
2. The sulphuric acid remaining after ignition, according to the extraction method and gentle heating of the residue, and perhaps also.
3. The sulphuric acid present as ammonium sulphate, by determining the amount of ammonia present. The difference between the total sulphuric acid and the amount of combined sulphuric acid found under 2 and 3 gives the amount of free sulphuric acid.

The method of A. Wuensch, for the determination of free sulphuric acid in leather, would also be of good service in this case.

For tanning purposes, lactic acid should further be free from hydrochloric acid, oxalic acid and iron, and therefore should not give with nitrate of silver in presence of nitric acid, nor with calcium sulphate solution, a precipitate, nor give with ferrocyanide of potassium a blue coloration.

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A NEW AND ACCURATE METHOD FOR THE DETERMINATION OF FREE ACID IN TAN LIQUORS.

By A. W. Hoppenstedt.

As all the present methods in use, including the Official Method, for the determination of free acid in tan liquors, give decidedly inaccurate results, the author has spent considerable time in devising a new method which would in the first place be accurate, and secondly, rapid and easy to manipulate. The result of my work is the following new method which I will call the "Quinine Method."

Place 50 cc. of the liquor in a 500 cc. flask and make up to the mark with water. To 200 cc. of diluted liquor add 20 cc. of quinine solution, mix thoroughly and filter. Take 100 cc. of the filtrate and titrate with N/10 KOH or NaOH, using phenolphthalein as indicator. Multiply the cc. used by 0.066 to obtain direct per cent. of acid, as acetic acid, in the original liquor.

The quinine solution is made up by dissolving 15 gms. of pure quinine in 110 cc. of 95% alcohol, which has been previously neutralized. To this add slowly, while stirring 90 cc. of water so as to make the volume 200 cc.

This method is based on the fact that certain basic alkaloids precipitate tannin and form soluble salts with the free acids. These soluble salts react acid to phenolphthalein, and so can be titrated as if the acid were in the free state. When titrating, the alkaloid is thrown out in the free form again as a white, insoluble precipitate.

This method can be applied to any tan liquor and using any dilution desired, and can also be applied direct on the strong liquors without any dilution, providing the amount of quinine used is increased in proportion. The amount of quinine used should always be sufficient to make the solution, after filtering, slightly alkaline to litmus, as otherwise if any free acid still were present, the same would dissolve part of the precipitate of quinine and tannin and tend to darken the solution when titrating.

The writer recommends to use the same solution used for the analysis of the liquor as this gives the best results, and where many liquor analyses are made daily a considerable amount of time is saved.

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In order to prove the accuracy of this method a 21° Bark. sapped oak liquor, a 18° Bark. sapped hemlock liquor and a 12° Bark. oak tail handler liquor were taken, the free acid determined in the same, and then 0.25% of a mixture of lactic and acetic acid added to them, and the free acid determined once more. The difference of the two titrations, after allowing for the dilution, would give the amount of acid added. In each case the results obtained agreed with the amount of acid added, which tends to prove the method to be correct.

DRY CHROMED HIDE-POWDER.

By H. C. Reed.

The "Chromed Hide-Powder Question" has recently taken on renewed life through the efforts of Mr. Ferdinand Kopecky, who discusses the problem at some length in the columns of the "Collegium," Nos. 199 to 203, inclusive. Although we may not all agree with the conclusions reached, yet we cannot but admire the conscientious research that leads up to them.

During a visit that Mr. Kopecky paid us last Summer, he left the writer some of his dry chromed powder, requesting that the results of the tests be given him. For this reason no criticism of the powder appeared in the Referee report last year. But inasmuch as Mr. Kopecky quotes from the writer's letter to him (Collegium No. 200, page 105) the entire letter is here published that there may be no doubt as to the actual outcome of the test.

Stamford, Connecticut, U.S.A., November 13th, 1905.

Mr. Ferdinand Kopecky,
Herolds Institute,
Leeds, England.

My Dear Sir :—

Owing to the large amount of work I have had to do as Referee this year, I have been unable, up to the present time, to inform you of the results from your chromed hide powder.

I regret very much to be obliged to report that this powder does not work at all well with the A.O.A.C., or shake method.

My results were as follows :

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	Kopecky	Wet Chromed
Solid Quebracho Extract	15.93	10.56
Chestnut "	11.93	11.26
Oak "	21.91	20.45
Hemlock "	24.38	20.41
Spent Oak Liquor.....	.42	.42
" Hemlock Liquor.....	.89	.88

On the extracts it was impossible to obtain clear non-tannin filtrates, while on the contrary the non-tannin filtrates from the liquors were entirely clear. This arouses a suspicion in my mind that your hide-powder is not sufficiently acid and that the clear filtrates with the liquors was due to the acidity of same. A test of the hide-powder itself showed that it was practically neutral and I am convinced that hide-powder should be slightly acid that good results may be obtained by the shake method. How this works with the filter-tube method I am not prepared to say.

Our Association has had some experience this year with dry chromed hide-powder, one not requiring washing. I remember that I showed you a sample of this hide-powder when you were here. The members who, immediately upon receipt of the sample, made their analyses, obtained fairly good results, while those who delayed for several weeks showed extremely poor results, accounted for only by deterioration in the quality, probably owing to a hardening and consequent failure in detannizing properties. This hide-powder was not a fully chromed powder, but chromed only slightly more than is the wet powder by the official method. Nevertheless, the point is interesting, and the keeping properties of even a fully chromed powder should be fully investigated before adopting it.

I believe that Mr. Teas has been working along the lines you recommended, but I have not been informed as to the results he obtained.

I will be much pleased to test further samples of your hide-powder, and to report back to you my findings. I enclose you your paper on the chromed hide-powder question, which has not left my hands, and it is not my intention to present the results of your hide-powder at our meeting, other than to say that chromed hide-powder from another source has been tested with negative results.

Regretting that you were unable to call upon me before your return to England, and trusting that I may have the pleasure of both seeing and hearing from you again, I am

Yours very cordially,

H. C. REED.

Duplicate tests were made with the powder, using three of the extracts mentioned in the latter, with the following results:—

Solid Quebracho Extract	14.68%
Chestnut "	13.50%
Oak "	22.00%

These results would indicate that duplication is impossible with the powder. Most certainly the sample, as furnished, could not

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under any circumstances be made to give clear non-tannin filtrates with extracts by our shake method.

Mr. Kopecky has made a rather exhaustive study of the effect of acidity upon the hide-powder and resulting non-tannins, as suggested in the letter quoted, comments upon which will be undertaken later in this article.

As to the manufacture of dry chromed hide-powder, the writer hastens to express his entire ignorance of the subject, and he will concern himself merely with his experience in the laboratory with such powders, and wherein they failed of being perfect.

A dry chromed powder to meet the requirements should have the following qualifications:—good absorption, uniformity in various lots, invariability after manufacture or unchangeableness upon keeping, and insolubility, that will enable the powder to be used dry. The latter qualification may cause comment, but much of the value and *raison d'etre* of a dry powder will be lost if we are compelled to soak or wash and use it with a moisture correction. The advantage over our present conditions would hardly make it worth the while.

Let it be said here, that there has but one powder been tried in the writer's laboratory that practically complied with all the conditions named, and that was the first tested, made by Mr. George P. Craighill, at the year of birth of chromed hide-powder; and Mr. Craighill informed the writer that the cost of production of the powder well-nigh precluded its use.

The powder furnished by the Referee last year was disappointing, but instructive. Here we had a powder that when freshly-made gave excellent results, but time proved it lacked in keeping qualities. Mr. Alden's heavily chromed dry hide-powder required soaking and washing, with wh exception it gave good results. There has recently been tested in the writer's laboratory a powder, requiring soaking and washing, but which would not give clear filtrates with extracts.

Mr. Kopecky says (Collegium No. 119, page 100), "The reduction of absorbent power seems to be entirely dependent on physical phenomenon, and especially on the diminished swelling properties of the powder. Over-chroming and over-neutralizing can cause the same————," and later, (Collegium No. 200, page 106), "Neutralized chromed powder absorbs more than pow-

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der which is slightly acid, and so does powder which is chromed heavier,—up to a certain degree, a great amount of chrome will, as mentioned before, reduce considerably the absorptive properties."

It is not to be disputed that with diminished swelling properties of powder we get diminished absorption and *vice versa,* and the writer is strongly of the belief that this is entirely due to a physical condition controlled by the degree of acidity. Although Mr. Kopecky states that a neutral chromed powder will absorb more than an acid powder chromed to the same extent, such has never been the experience in our laboratory.

The sample of hide-powder made by Mr. Alden was very heavily chromed, yet it did not seem to lose its absorptive properties to a noticeable extent as might be expected from Mr. Kopecky's statement above quoted. On the other hand, Mr. Kopecky's powder, not nearly so heavily chromed as Mr. Alden's, would not approach the latter in absorptive capacity. This was readily explained when the two powders were tested for acidity, Mr. Kopecky's showing a neutral reaction and Mr. Alden's a decided acid reaction. Thus it would appear that whether a powder is unchromed, lightly chromed or heavily chromed, the degree of acidity determines its power of absorption. Therefore, it follows that the wet chromed powder used in our official method should be as uniformly acid as possible to give the greatest concordance, and this applies as well to a dry chromed powder.

Mr. Kopecky shows comparative tests of the filter tube, shake, maceration and stirring methods, very much to the detriment of the first mentioned. It is to be hoped that his results may help to induce the I. A. L. T. C., to abandon their present official method. Dry chromed powder was used in these experiments and the low non-tannins from the filter tube method are explained by the fact that the tannin is absorbed by a limited quantity of the powder, gallic acid set free and absorbed by fresh, unacted-upon powder. When hide-powder is added in separate portions to a tannin solution the same effect is produced to a lesser degree, and those who have in the past used the cotton-plugged funnel and after shaking throw the powder and all into the funnel and returned till clear, tamping the hide, if necessary, will recall that lower non-tannins were obtained than by the present method of paper and kaolin,

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undoubtedly for the very reason given above. Mr. Kopecky's method of throwing upon a filter the macerated or shaken solution, allowing to run through and returning twice (Collegium No. 200, page 104), may be justly criticised on the same grounds. The objection to the filter tube method in this country has always been based on the fact of detannization by a limited quantity of powder and absorption of non-tannins by the unacted-upon powder.

Returning again to the subject of the acidity of powders, the writer confesses his inability to account for the fact that in making his experiments upon acidulation of hide-powders (Collegium No. 201, page 112), Mr. Kopecky abstained entirely from using acetic acid. He finds that lactic and sulphuric acids are not sufficiently volatile and formic too volatile. Later he employs acetic in acidulating tannin solutions and his results prove it the best. His theory that tannic acid is liberated from the various glucosides of the tannins by the presence of an acid, either in the hide-powder or added to the tannin solution, the tannic acid being rapidly absorbed, in contra-distinction to the tannin which is not, —is ingenious. His conclusion as to the necessity of having an acid condition of either powder or solution bears out the views expressed by the writer. However, the acidulation of the entire solution, an idea suggested by Mr. Mardick in his collaborative work for our Association last year, seems a step in the wrong direction. Mr. Kopecky in reality condemns the method himself when he makes the following statement, used in another connection, (Collegium No. 202, page 124), "I am far from stating that the whole quantity of 'insolubles' is due to fermentation, but think I have clearly proved that on acidulating tannin solutions a great percentage of tanning matter is converted into 'insolubles,' that at the same time the percentage of non-tannins increases and the color of the acidified tannin solution is greatly improved." The right of a chemist to tamper with a sample of extract by adding acid, increasing insoluble and non-tannins, is questionable to say the least, but granting it is permissible, the addition of the acid will plunge us into another difficulty, *viz:*—the impossibility of making filtration for soluble solids at a point where the acid has ceased its precipitation of insolubles.

As much as a year ago experiments were made in the writer's

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laboratory upon acidulating tannin solutions immediately prior to shaking. The hide-powder was our official, wet chromed powder, and varying quantities of the acid (Acetic) apparently produced no alteration in the non-tannin content. Possibly this would not have been the case if a neutral chromed powder had been employed, but the result arouses a suspicion, however, that an acid chromed powder and a neutral powder with acidulated solution are not of necessity identical in effect. Provided we ultimately attain the goal of our ambition, a powder that can be used dry, it would be far preferable to have the acidity in the powder than to be obliged to add it to the solution, thereby necessitating a "correction."

Mr. Kopecky recommends that the present I. A. L. T. C. method be abandoned and what the writer presumes to mean our method, *not* adopted, but a method perfected from his recommendations substituted in its place. This truly seems a pity, inasmuch as the difference between our official method and the method worked out by Mr. Kopecky is so slight.

His alternative recommendations relative to acidity are:—

(1) Treat hide-powder with definite quantity of acetic or formic acids.

(2) Add definite quantity acetic acid to solution immediately prior to shaking.

(3) Add definite quantity acetic acid to entire solution prior to analysis.

Thus this first recommendation, if adopted, would mean that the I. A. L. T. C., would use an acid lightly chromed hide-powder, and that is exactly what we are using here, and Mr. Kopecky's own figures show that there is but little difference between the shake and the maceration methods.

Surely the work of our Association last year showed one thing very clearly, that in the hands of the careful analyst (and methods are not made for the careless), hide-powder, chromed and used as directed by our official method, gives concordant results.

It is suggested to our confreres of the I. A. L. T. C. that having approached thus nearly to our method in their individual investigations, they unite and adopt our method as it stands. Then, if there are minor points to decide, whether of possible advantage in dry chromed hide-powder, of methods of filtration, etc., etc.,

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we can work in double harness, pulling together, accomplishing much.

And one thing more. Should our brothers across the water decide to adopt our method, they would be received with a welcome, harbouring no ill-timed elation, but a welcome as honest in intent and feeling as these remarks are meant to be.

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EDITORIAL

In this issue appears a copy of a letter which will be read with interest and satisfaction by American leather chemists.

The action of the German Section of the I. A. L. T. C., in officially endorsing the use of chromed hide-powder is remarkable, only because of its independence.

Official European recognition of the merits of chromed powder was to have been expected sooner or later; but when such recognition and endorsement comes from a single section, independent of the action and between the meetings of the International organ-

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ization, there must needs be small doubt in the minds of the German chemists as to its wisdom.

We congratulate our Teutonic brethren upon having taken the initiative in this step toward concordance, and trust that they will pardon a manifestation of human nature, when we remind them that the American chemists have for five years enjoyed a peace of mind on the subject of non-tannin results, that would have passed all understanding in our days of unchromed hide-powder.

We anticipate the time in the near future, when the American and European methods of tannin analysis will be identical; and after the adoption of chromed hide-powder, the abandonment of that accumulator of non-tannins—the filter tube—is appropriately in order. Hope is born of faith, and until our faith in the shake method fails, we shall continue to hope for an international method that is also intercontinental.

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CHROMING OF HIDE POWDER.

(Report of Sub-Committee for 1906).

By C. W. Norris.

I regret to state that the committee appointed to investigate the Chroming of Hide Powder found it impossible to carry out all the work outlined by our Referee.

I also regret that it was impossible to obtain Chromed Hide Powder from Dr. Kopecky in time for the work. This committee consisted of Messrs. F. W. Alden, W. H. Teas, F. W. Ackerman, C. Drueding and C. W. Norris, Chairman. Mr. Teas was unable to do any laboratory work. However, Mr. Teas furnished the committee with a quantity of Chromed Hide Powder, which was prepared by the Kopecky method. The chairman also prepared a quantity of Chromed Hide Powder, which was prepared by the Kopecky method, but regret to say that a sufficient quantity for the whole committee could not be prepared in time for the work. The following directions were issued:—

Analysis of Extracts.

Quebracho Extract	Dilution	6 g. per liter.
Hemlock Extract	"	14 " " "
Chestnut Oak Extract	"	15 " " "

Complete analysis of each sample by official method adopted November, 1905.

STUDY I.

Non-Tannin Determination Using the Kopecky Hide Powder.

I would suggest that these determinations be made upon the solutions which remain after making complete analysis. A quantity of Hide Powder sufficient for three analyses shall be prepared in the following manner:—Digest with 25 times its weight of water till thoroughly soaked. Agitate by stirring occasionally for several hours and let stand over night. Wash, test for sulphates and press as in Official Method. Squeeze so that the wet hide will contain between 70-75% of water. Use approximately 20 grams of wet hide for moisture determination. Add to 200 cc. of the Extract Solution such quantity of the wet hide as represents from 12-13 grams of dry hide.

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STUDY II.

Three Chrome liquors were prepared as follows:—

LIQUOR NO. 1.

100 g. Chrome Alum
6 g. Sodium Carbon (dry) } 1000 cc. water.

LIQUOR NO. 2.

100 g. Chrome Alum
10 g. Sodium Carbonate } 1000 cc. water.

LIQUOR NO. 3.

100 g. Chrome Alum
14 g. Sodium Carbonate } 1000 cc. water.

Non-tannin determination upon each of the three extracts, using the regular hide powder chromed with the Chrome Liquors Nos. 1, 2, and 3, in the following proportions,—5, 10 and 20% of Chrome Alum per gram of dry hide.

CHROMING WITH LIQUOR NO. I.

(A) A quantity of hide powder sufficient for three analyses shall be prepared as follows:—Digest with 25 times its weight of water. Add 0.5 cc. (or 5% Chrome Alum) of Chrome liquor for each gram of hide taken. (Add in two portions: one-half at the beginning and the other half two hours later). Agitate occasionally for several hours and let stand over night. Wash, test for sulphates and press to have 70-75% of water, as in Official Method. Use 20 grams of wet hide for moisture determination. Shake 200 cc. of Extract Solution with such quantity of the wet hide as represents from 12 to 13 grams dry hide.

(B) Weigh off another quantity of hide as in *(a)* and add 1 cc. (or 10% Chrome Alum) of Chrome Liquor for each gram of Hide Powder. (Adding Chrome Liquor in two portions as in *(A)*).

(C) Weigh off a third portion of hide as in *(A)* and add 2 cc. (or 20% Chrome Alum) for each gram of hide powder. (Adding Chrome Liquor in two portions as in *(A)*).

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CHROMING WITH LIQUOR NO. II.

Proceed exactly the same as with Liquor No. I.

CHROMING WITH LIQUOR NO. III.

Proceed exactly the same as with Liquor No. I.

Remarks.

State percentage of water in wet hide and amount of wet hide taken in each case.

Note the clearness on non-tannin filtrates.

Note the amount of washing necessary to free the hide from sulphates.

Note whether there is much chrome liquor left in the solution when hide is ready for washing.

Results of the work are shown in the following tables:—

Quebracho Extract.

Complete Analysis by Official Method	Norris	Alden	Ackerman	Drueding	Average
Water	13.11	13.63	12.67	12.72	13.08
Total Solids	86.89	86.37	87.33	87.28	86.92
Sol. Solids	86.62	84.89	86.88	86.60	86.25
Insolubles	.27	1.48	.45	.68	.67
Non-tannins	14.71	14.84	15.17	15.40	15.03
Tannins	71.91	70.05	71.71	71.20	71.22
Wt. Wet Hide gms.	45	47	50	50	
Per cent. H_2O in wet hide	72.88	72.18	74.50	....	....
Dry Hide, grams	12.75	13.1	12.75	12 to 13	....

Hemlock Extract.

Complete Analysis by Official Method	Norris	Alden	Ackerman	Drueding	Average
Water	47.96	48.15	47.43	47.81	47.84
Total Solids	52.04	51.85	52.57	52.19	52.16
Soluble Solids	48.20	49.13	49.35	48.41	48.77
Insolubles	3.84	2.72	3.22	3.78	3.39
Non-Tannins	23.28	23.40	24.01	23.93	23.65
Tannins	24.92	25.73	25.34	24.48	25.12
Wt. Wet Hide, grams.	45	47	50	50	....
Per Cent. H_2O in Wet Hide	72.88	72.18	74.50	....	....
Dry Hide, grams	12.75	13.1	12.75	12–13	....

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Chestnut Oak Extract.

Complete Analysis by Official Method	Norris	Alden	Ackerman	Drueding	Average
Water	58.74	59.11	58.28	58.53	58.67
Total Solids	41.26	40.89	41.72	41.47	41.33
Soluble Solids	40.96	40.52	41.30	41.17	40.99
Insolubles	.30	.37	.42	.30	.35
Non-Tannins	19.72	19.86	19.81	18.97	19.59
Tannins	21.24	20.66	21.49	22.20	21.39
Wt. Wet Hide, grams	45	47	50	50	....
Per Cent. H_2O in Wet Hide	72.88	72.18	74.50	....	....
Dry Hide, grams	12.75	13.1	12.75	12–13	....

STUDY NO. I.

Non-Tannins Using Kopecky Chromed Hide Powder.

	QUEBRACHO		HEMLOCK		CHESTNUT OAK	
	Hide prepared by		Hide prepared by		Hide prepared by	
	Teas	Norris	Teas	Norris	Teas	Norris
Norris	13.89	14.53	22.57	23.59	19.79	19.19
Alden	14.61	16.34	22.72	26.42	19.69	19.62
Ackerman	15.05		24.12		20.15	
Drueding	14.02		22.33		16.89*	
Average	14.39		22.93		19.88	

* Omitted from average.

The non-tannin filtrate (where each hide powder was used) of the Quebracho and Hemlock Extracts was slightly turbid, while that of the Chestnut Oak was perfectly clear.

Greater variation of non-tannins is found than by the Official Method. Since these results were obtained with the hide powders made by Messrs. Teas and Norris, the chairman has received from Dr. Kopecky, of Leeds, England, a quantity of Chromed Hide Powder, and would suggest that it be given a trial in the general collaborative work.

STUDY NO. II.

Non-Tannins.

Regular Hide Powder chromed with 5, 10 and 20% of Chrome Alum of varying basicity.

Chromed with 5% Chrome Alum	QUEBRACHO			HEMLOCK			CHESTNUT OAK		
	No. 1	No. 2	No. 3	No. 1	No. 2	No. 3	No. 1	No. 2	No. 3
Norris	14.84	14.84	14.79	23.25	23.25	23.35	19.91	19.87	19.68
Alden	16.38	15.99	15.49	23.40	23.64	22.90	19.79	20.21	19.83
Ackerman	15.02	15.98	15.30	22.93	24.28	24.44	19.34	18.57	18.68
Drueding	15.44	15.01	15.79	24.72	22.81	23.85	18.70	18.76	18.98

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Chromed with 10% Chrome Alum	QUEBRACHO			HEMLOCK			CHESTNUT OAK		
	No. 1	No. 2	No. 3	No. 1	No. 2	No. 3	No. 1	No. 2	No. 3
Norris	15.03	14.54	14.80	23.49	23.27	23.20	20.08	19.91	19.81
Alden	14.03	14.66	14.74	22.86	22.97	23.04	19.94	19.78	19.82
Ackerman	14.55	14.85	14.45	23.27	23.15	23.57	18.63	18.77	18.49
Drueding	14.41	14.38	14.95	23.27	21.89	23.42	18.36	18.46	18.95

Chromed with 20% Chrome Alum	QUEBRACHO			HEMLOCK			CHESTNUT OAK		
	No. 1	No. 2	No. 3	No. 1	No. 2	No. 3	No. 1	No. 2	No. 3
Norris	15.50	15.18	15.12	23.66	23.32	23.42	20.52	20.33	20.15
Alden	15.18	15.07	15.46	23.84	23.54	23.11	20.83	20.35	20.10
Ackerman	14.52	14.90	14.75	22.98	22.76	23.28	18.57	18.67	18.85
Drueding	14.41	14.33	14.64	23.03	21.85	22.85	18.42	18.49	18.65

The Chrome Alum is completely absorbed in chroming the hide in all three liquors where 5% is added. Where 10% is added a small excess of chromium is left after chroming in Liquor No. 1, with Liquor No. 2 only a trace, while with Liquor No. 3 it is all absorbed. Where 20% is added a considerable excess is left after chroming with Liquor No. 1, somewhat less with Liquor No. 2, and still less with Liquor No. 3.

The Hide Powder chromed with 10% shows a considerable degree of tannage, while with 20% the hide is fully chromed and will stand washing with water at nearly the boiling temperature without destroying its absorptive properties for tannin.

In making a study of the three tables of results the chairman is at a loss to account for the great variation of results among the several analysts and also of the results of the analysts themselves. In some cases the variations may be due to insufficient washing of the hide powder or not squeezing to the proper moisture content. However, a careful study of the results of the Chairman (Norris) leads him to believe that the absorption of the hide powder *decreases* with *heavier* chroming, while there is an *increase* of absorption with the increasing *basic* property of the liquor.

Taking the chairman's results of each extract, where the hide powder was chromed with *10% of Liquor No. 2,* and comparing them with his non-tannins by the Official Method it will be seen that practically the same results were obtained. The non-tannin filtrates in all cases were clear and bright, in fact, more colorless than those by the Official Method.

It is difficult to draw any conclusion, as there has been so little work done. A chromed hide powder of this nature has no par-

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ticular advantage, unless it could be chromed in large quantities, thoroughly washed, air-dried, and when an analysis is to be made digest in water a few minutes to secure good absorption; then press to proper moisture content.

It is my opinion that a hide powder as fully chromed as this powder, will not deteriorate upon long standing. It is to be hoped that work along this line will be fully investigated.

COMPARATIVE ANALYSES WITH SPROUTED ALUMINA, CHROMED AND NON-CHROMED HIDE POWDER.

(Translated from the "Collegium," 1906, p. 158).

At the meeting of the German Section of the I. A. L. T. C., at Frankfort, a.M., on the 22d of April, it was decided to send the following communication to the Executive Committee of the I. A. L. T. C.:—

MR. G. SCHWEITZER, PRESIDENT I. A. L. T. C.,
ROZTOK, BOHEMIA:—

We herewith inform the Honorable Executive Committee that at the meeting held on the 22d of April, at Frankfort, a.M., the German Section unanimously adopted,

"To use from now on, for the purpose of tannin analyses, a slightly chromed hide powder containing from .4 to .5% Cr_2O_3, instead of the usual slightly acidified hide powder made by the firm of Mehner & Stransky, in Freiberg. Through the efforts of the German Section this new hide powder will be made by the "German Experiment Station for Leather Industry" at Freiberg i.Sa., and can be obtained from them."

This decision was reached under the following circumstances:

The German Section appointed at the meeting on the 25th of April, 1905, at Frankfurt, a commission composed of Messrs. Klenk, Paessler and Philip to look into the question of obtaining a sufficient amount of a good hide powder from the firm of Mehner & Stransky in Freiberg. This commission started negotiations, the same being given in "Collegium" (1905, p. 245, 277 and 279). The German Section wanted to obtain an amount of

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hide powder from the said firm sufficient to cover its need for one year and desired the same distributed and forwarded through the "German Experiment Station for Leather Industry," or its Executive Board.

Everything had been arranged to give the members a uniform hide powder for one year, when it was found impossible to obtain in advance the yearly requirements of the powder from the firm of Mehner & Stransky. The firm claimed that it was unable to make more hide powder than was used on an average, and consequently could not supply larger quantities for storing. At the meeting of the German Section on the 3d of December, 1905, these conditions were fully discussed. Dr. Paessler was requested,as at former meetings, to have the hide powder made by the Experiment Station. Although Dr. Paessler was formerly opposed to this proposition, he intimated that he would look into the matter, as he had also arrived at the conclusion that, that which the German Section strove to obtain could not be gotten from the firm of Mehner & Stransky. At the same meeting Dr. Paessler also stated that he had again been occupied with the question of using a slightly chromed hide powder for the filter-tube method and that he had obtained satisfactory results. With such a hide powder about the same non-tannins were obtained as with the ordinary hide powder. In the meantime Dr. Paessler has occupied himself with the production of a slightly chromed hide powder and obtained good results in every way. At the past meeting held on the 22d of April of this year he announced that it is now possible to manufacture a sufficient amount of this hide powder which will remain constantly uniform, so that the demand to produce a large uniform supply to be kept on hand, is now possible. According to Dr. Paessler, there is now stored in the Experiment Station about 60 kg. of hide powder ready for use and soon 40 kg. more will be ready. Accordingly, there is more on hand than the yearly requirements of the German Section, so that undoubtedly it will also be possible, in the near future, to meet the total requirements of the foreign sections for chromed hide powder from Freiberg. In our opinion such an arrangement in the interest of a uniform material for tannin analysis would be welcomed by the I. A. L. T. C. Consequently, it can be left to the I. A. L. T. C. to decide later whether they wish to accept this

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slightly chromed hide powder or a stronger chromed hide powder.

After Dr. Paessler had made larger quantities of this slightly chromed hide powder, he communicated with several associates and placed large samples at their disposal. Several German chemists used this opportunity to test this hide powder at the same time when doing the comparative tests with the "Alumina Method" of Wislicenus, as directed by the German Section. The results have been tabulated by Dr. Becker and were critically discussed at the meeting on the 22d of April. The tabulation of the results and the report of Dr. Becker on the same, will be published later in the "Collegium." Annexed we enclose the former. The conclusions to be drawn from these results are, that this slightly chromed hide powder gives on an average better comparative values than any other preparations tried in the past and that the non-tannins obtained are about the same as those obtained with a good ordinary hide powder. In the latter case the results are occasionally a little higher. This is probably due to the absorption of the ordinary hide powder not being as regular and often containing more soluble matter, which increases the amount of non-tannins. It consequently can be accepted, that the results obtained with chromed hide powder are the correct ones.

Dr. Paessler has further determined by experiments that grape-sugar, cane-sugar and dextrin are not absorbed by chromed hide powder. The chemists who have made tests with the slightly chromed hide powder all speak very favorably of the same and point out as benefits derived, that it gives a greater uniformity of results and swells up very little in the filter tube which greatly lessens the work with this hide powder. Also that it contains less soluble matter (about 2-3 mg. in 50 cc. of the filtrate after discarding 30 cc.). Foreign chemists, especially Messrs. Jedlicka and Boegh, who have done considerable work with it, have also expressed their satisfaction with this hide powder, and the possible introduction of the same as a decided step in advance.

After the benefits of the slightly chromed hide powder were recognized at the last meeting of the German Section, complaints were made of the poor quality of the hide powder recently furnished by Mehner & Stransky. Mr. Schorlemmer reported, for instance, that a hide powder recently obtained, (5 gms. hide powder) contained 16.5 mg. of soluble substances (in 50 cc. of solution) and that the non-tannin filtrate had a gluey appearance.

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The members of the "Society of German Color and Tanning Extract Manufacturers" who were present at the meeting, greatly lamented the unreliability of the analyses made with such a hide powder and pointed to the growing dissatisfaction in commerce and industry, by the repeated changes in the method of analysis, and also by the past insufficient shipments of hide powder of varying composition.

These undeniable evils and justified complaints were the reasons for the motion to be made to use the slightly chromed hide powder at once. As already stated this motion was unanimously carried. It was further decided to notify the Executive Committee of this, giving the full reasons and in order to remain just to the past resolutions of the I. A. L. T. C., it was decided from now on to add to the analyses reports the note, "By the use of slightly chromed hide powder according to the resolutions adopted by the German Section on the 22d of April, 1906."

We note again that the following facts are the reason for our resolutions:

1. The unsuccessful attempt to obtain from the firm of Mehner & Stransky a supply of hide powder to be kept in store and sufficient for the German Section for one year;

2. The poor quality of the last shipments of hide powder from the firm of Mehner & Stransky;

3. The good quality of the hide powder made by the "German Experiment Station for Leather Industry;

4. The certainty of the delivery of larger amounts of uniform hide powder by the "German Experiment Station for Leather Industry".

Before the coming conference in September at Prague, more results will be on hand on this preparation. We will then make the motion to have this hide powder universally used and hope that thereby a decided advance will again be achieved in the efforts to obtain uniform results.

Very respectfully the "German Section".

By the Executive Board,

DR. H. BECKER,	DR. PAESSLER,	PROF. DR. PHILIP,
President.	*Secretary.*	*Treasurer.*

MAY, 1906.

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AVERAGES OF TABLES.

	Ordinary hide-powder acidified with Citric Acid. %	Al_2O_3. %	Chromed Hide-Powder. A. Slightly chromed. %	B. Strongly chromed. %	C. Strongly chromed, part. neu. %
SOLID QUEBRACHO EXTRACT					
Highest Tannin	73.4	76.9	76.0	75.4	76.0
Non-Tannins	6.4	4.3	5.2	4.6	4.0
Lowest Tannin	67.07	68.00	72.0	73.4	74.2
Non-Tannins	4.0	2.2	3.0	3.2	3.7
Average Tannin	71.6	74.9	74.1	74.7	75.4
Non-Tannins	5.4	2.9	4.2	4.1	4.0
Difference between highest and lowest Non-Tannins.	2.4	2.1	2.2	1.4	0.3
OAK WOOD EXTRACT					
Highest Tannin	22.3	27.9	24.6	24.6	24.6
Non-Tannins	14.9	12.5	12.1	12.4	12.1
Lowest Tannin	20.5	23.2	22.9	23.1	23.5
Non-Tannins	12.7	7.8	10.8	10.8	10.7
Average Tannin	21.2	25.6	23.9	23.4	24.1
Non-Tannins	14.2	9.9	11.5	11.5	11.1
Difference between highest and lowest Non-Tannins.	2.2	4.7	1.3	1.6	1.4
HEMLOCK EXTRACT					
Highest Tannin	23.4	27.0	24.4	24.6	25.2
Non-Tannins	14.6	12.4	13.1	13.3	12.9
Lowest Tannin	16.6	18.8	23.2	22.0	23.3
Non-Tannins	13.0	8.9	12.2	12.4	11.7
Average Tannin	21.4	24.8	23.8	23.4	24.5
Non-Tannins	13.8	11.2	12.7	13.2	12.1
Difference between highest and lowest Non-Tannins.	1.6	3.5	0.9	0.9	1.2
SUMAC EXTRACT					
Highest Tannin	22.7	29.3	25.2	25.4	27.9
Non-Tannins	21.9	13.2	17.3	16.6	15.7
Lowest Tannin	18.0	26.7	23.9	25.1	25.9
Non-Tannins	18.9	11.6	16.4	16.0	13.8
Average Tannin	20.6	28.4	24.6	25.3	27.0
Non-Tannins	20.3	12.6	16.7	16.3	14.6
Difference between highest and lowest Non-Tannins.	3.0	1.6	0.9	0.6	1.9

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	Ordinary hide-powder acidified with Citric Acid.	Al_2O_3	Chromed Hide-Powder A Slightly Chromed	B Strongly Chromed	C Strongly Chromed part. neu.
	%	%	%	%	%
MANGROVE EXT.					
Highest Tannin	37.9	40.1	38.7	38.2	38.5
Non-tannins	8.5	6.4	7.8	10.5	7.8
Lowest Tannin	35.9	38.2	37.5	33.9	37.3
Non-tannins	6.5	5.0	5.7	7.5	7.2
Average Tannin	36.7	38.9	37.8	36.8	37.9
Non-tannins	7.9	6.0	7.1	8.5	6.7
Diff. betw. highest and lowest non-tannins	2.0	1.4	1.9	3.0	0.6
MIMOSA EXT.—RENNER IDEAL					
Highest Tannin	31.8	34.5	32.9	33.2	33.4
Non-tannins	6.7	5.4	5.4	5.4	5.9
Lowest Tannin	30.6	32.1	31.9	32.1	31.7
Non-tannins	5.7	3.1	4.9	5.0	4.8
Average Tannin	31.3	33.1	32.5	32.7	32.6
Non-tannins	6.2	4.5	5.2	5.2	5.1
Diff. betw. highest and lowest non-tannins	1.0	2.3	0.5	0.4	1.1
MIMOSA EXT.—RENNER D.					
Highest Tannin	39.6	43.4	41.0	41.1	42.1
Non-tannins	11.4	8.2	9.7	9.2	9.8
Lowest Tannin	38.6	41.8	39.9	40.9	40.3
Non-tannins	10.3	7.1	9.0	8.8	8.0
Average Tannin	39.0	42.4	40.7	40.7	41.7
Non-tannins	10.9	7.7	9.3	8.9	8.8
Diff. betw. highest and lowest non-tannins	1.1	1.1	0.7	0.4	1.8
MIMOSA EXT.—REGULAR LIQUID					
Highest Tannin	33.2	36.2	34.8	34.3	35.1
Non-tannins	10.5	7.6	10.2	9.8	9.8
Lowest Tannin	31.7	34.0	32.0	32.1	32.1
Non-tannins	9.7	6.9	8.3	8.8	8.0
Average Tannin	32.1	35.0	33.1	33.1	33.5
Non-tannins	10.0	7.2	9.3	9.3	9.0
Diff. betw. highest and lowest non-tannins	0.8	0.7	1.9	1.0	1.8

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ANALYSIS OF COMMERCIAL ACIDS.

(Report of 1906 Sub-Committee).

By W. H. Teas.

The following instructions were issued to the members of our committee:

"As Chairman of the A. L. C. A. Committee on ANALYSIS OF COMMERCIAL ACIDS, I am sending you samples of commercial acids, which please analyze according to the following directions, and report as promptly as possible:

LACTIC ACID:

Total Acidity.

Dilute 10 grams to 500 cc. and titrate 100 cc. with N/10 NaOH.

Volatile Acids.

Dilute 10 grams to 100 cc., distill off 90-95 cc., and save distillate; make up residue to 100 cc., and again distill off 90-95 cc., combine the two distillates and make up to 200 cc. Take 100 cc. of this, add 40 cc. of 1% permanganate solution, and distill off 90-95 cc., make up this distillate to 200 cc. and titrate. Result double gives volatile acids.

Free Sulphuric Acid.

Add 10 grams of the acid to 90 cc. of 98% alcohol and let stand (corked) over night at 150°-160° F.; filter, washing with 90% alcohol. Add 50 cc. water and ½ gram sodium carbonate to the filtrate, and boil off or recover the alcohol; acidify the *aqueous* residue with HCl and determine the sulphuric acid gravimetrically.

Lactic Anhydride.

Weigh 5 grams of acid, titrate in 500-600 cc. water with N/2 NaOH, take reading for total acidity. When end point is reached add 20 cc. additional alkali, bring to boil and boil ten minutes; then add 20 cc. N/2 acid and titrate with alkali for second end point; reading will give percentage of lactic anhydride.

ACETIC ACID:

Determine total acidity, volatile acids and free sulphuric acid (if present) according to methods for lactic acid.

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Free Mineral Acids.

Twenty-five grams of the acid is mixed with a known quantity of N/10 NaOH, evaporated, ignited at a low red heat. Cool and add same amount of N/10 acid as was used of N/10 soda; heat to expel CO_2, filter, washing residue, and titrate filtrate and washings with N/10 NaOH. Volume so used equals the amount of mineral acid in 25 grams of the acetic acid[1].

OXALIC ACID:

Determine Total Acidity by Titration.

Dissolve 5 grams in a liter of water, filter on a dry filter; take 100 cc., neutralize with ammonia, precipitate with calcium chloride solution, wash thoroughly by decantation, add sulphuric acid, heat, and titrate with permanganate.

Determine Ash, and if appreciable, titrate with N/10 acid and litmus or methyl orange.

FORMIC ACID:

Determine Total Acidity by Titration.

Dilute 5 grams to 500 cc. and take 200 cc. for distillation. Distill until no acid comes over and titrate the distillate. If necessary, distill the 200 cc. until 175 cc. have gone over, then cool, add 200 cc. water to residue and continue the distillation.

Determine formic acid as follows: Dilute 5 grams to 500 cc., take 100 cc., make alkaline with sodium carbonate, warm and add an *excess* of standard permanganate. Acidify with sulphuric acid and run in a measured volume of oxalic acid until the precipitate is dissolved and the color disappears. Titrate the excess of oxalic acid with permanganate. A volume of oxalic acid equal to that used is titrated with permanganate, and this result subtracted from the total amount of permanganate used will give the amount of permanganate required to oxidize the formic acid[2].

The Chairman will welcome results by other methods than the above, but please do not omit the above methods."

The results of the analyses of the different acids appear on the accompanying table:

[1] Sutton, 9th Edition, 90.
[2] Sutton, 9th Edition, 108.

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AMERICAN LEATHER CHEMISTS ASSOCIATION

COMMITTEE ON COMMERCIAL ACIDS

Committee Results on Samples Distributed by the Chairman

LACTIC ACID				ACETIC ACID		OXALIC ACID			FORMIC ACID				
Total Acidity	Volatile Acids	Free Sulphuric Acid	Lactic Anhydride	Total Acidity	Volatile Acids	Total Acidity (Titration)	By Precipitation	Ash	Total Acidity	Volatile Acid	By Jones' Method	Sulphuric Acid	ANALYST
19.91%	1.33%	1.32%	5.06%	27.19%	18.21%	96.31%	96.19%	nil	84.09%	79.55%	95.07%	.02	A. A. Claflin
20.30	1.15	2.30	5.63	27.86	15.23	100.07	100.17	....	85.50	82.10	84.80	....	C. Eachus
18.70	1.08	1.45	3.87	26.02	18.84	92.31	98.43	.05	78.71	72.33	76.94	.05	J. H. Russell
20.32	1.11	....	5.59	27.94	13.57	99.08	97.44	.07	85.49	33.08	55.49	....	F. W. Alden
20.04	1.07	1.40	5.62	27.27	19.11	98.45	98.19	.05	83.99	83.11	83.34	trace	W. H. Teas

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Commenting on the work, Mr. Eachus writes: "I think the methods for Oxalic, Formic and Acetic Acids are good enough, if the method for volatile acids is changed to something sensible and practicable.——The method given in your directions for determining volatile acids in lactic acid is a very poor one. The permanganate does no good, and where 100 cc. of the combined distillates are diluted to 140 cc. with 1% permanganate, the volatile acid is not all regained by distilling off 90-95 cc. It would be much better to take 100 or 200 cc. of solution (10 gr. to 500 cc.) and distill this four or five times until all the volatile acid comes over."

Referring to the volatile acid determination, Mr. Alden writes: "The determination is a difficult matter. More or less of the acid is sure to be lost even with a delivery tube attached to the end of the condenser leading the distillate down into a flask with a cotton plug in the neck. Would it not be well to drive off volatile acids under certain prescribed conditions and titrate the residue?"

The chairman directed the use of the same method for the determination of volatile acid in acetic acid as in lactic acid, in order that a comparison of results by this method might be had on a non-volatile acid and on a volatile acid. It is evident that a method for determining volatile acid in lactic acid will probably not be suited for this determination in acids like acetic or formic, and *vice versa*. The determination of volatiles in lactic acid is at best, unsatisfactory, as the supposedly non-volatile lactic acid is found in the distillate, and numerous distillations will not always entirely remove acetic acid from the commercial lactic acid. Mr. Eachus writes me that he has some lactic acid giving absolutely no volatile acids on distillation. Experiments on a mixture of this acid with different amounts of acetic acid might lead to a more absolute method of determining volatiles. On the other hand, Mr. Claflin in his wide experience with lactic acid, has never found a sample from which a considerable quantity of volatile acid could not be distilled.

The results on volatile acid in lactic acid by the prescribed method are very satisfactory, much more so than was anticipated. The method was recommended by Mr. Claflin, who has probably adopted it because of its results, rather than from lack of objec-

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tion to the method. The effect of the flame temperature, particularly toward the end of the distillation when the volume is small, cannot be good. With small amounts of volatile acids present, the vapor lost through improper condensation will greatly influence the results. The idea of destroying, by means of the permanganate solution, the lactic acid in the first distillates is a good one.

A method for volatile acids in lactic acid, sent by the Referee, requires less time and trouble,—there can be no objection to temperature conditions, vapor losses are not factors in the results, but any lactic acid volatilized is counted as a volatile acid. The method is as follows: 100 cc. of the diluted solution are evaporated to 50 cc. three times over a steam bath, 50 cc. of water being added after each evaporation to make up to the original volume taken. After the third evaporation the cooled residue is titrated and the difference between this value and the total acidity is figured as volatile acids.

This method showed 1.18% volatile after the first evaporation, 1.78% after the second, and 2.14% volatile acids after the third evaporation in the committee sample of lactic acid. Are these high results due to the evaporation of some lactic acid, or to a more complete removal of the acetic acid? The steam-bath method appeals strongly to the chairman as a volatile method for lactic acid, for, in addition to the above noted advantages, it would appear that the method admits of greater possibility for uniform operation.

The determination of volatile acids in acetic and formic acids should also be conducted over the steam-bath or by distilling in a current of steam. By the latter method Mr. Russell found 25.7% volatile acid in the acetic acid, as against 18.84% by the method given for lactic acid. The chairman found 27.17% volatile in the acetic acid by evaporating to dryness on a steam-bath, and adding water the instant the evaporation was complete. The same treatment of the formic acid gave 83.84% volatiles, as compared with 83.11% by three distillations.

The method given for free sulphuric acid can be safely recommended, and it may be added that 98% alcohol has no advantages over 95% alcohol for this determination.

Undoubtedly the true acid values of the different acids are

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shown by the total acidity determined by direct titration. Except in the case of a solid acid containing water of crystallization, variation is due to variation in standard alkalies and to the personal error. All other determinations in acid analysis are, of course, for the purpose of detecting adulteration.

The wide variation in total acidity values for the oxalic acid sample emphasizes the importance of selecting normal crystals for analysis. With this acid the total acidity is peculiarly the true value, as the results from calcium precipitation will also include any oxalates present. The ash is only important in cases where the total acidity is low.

Jones' method for formic acid is rapid and satisfactory, but with high-priced acids there is always the temptation to adulterate and it is well to look for the presence of cheaper acids as a check on the specific acid determination.

As this is the first work the Association has undertaken in this line, the chairman feels neither qualified nor at liberty to make any definite recommendations as to methods, and trusts that the publication of the results of the committee's work will lead to further work more generally distributed, from the results of which we may be able to determine those methods worthy of being made official.

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THE DETERMINATION OF ACID IN TAN LIQUORS

(Report of 1906 Sub-Committee.)

By A. W. Hoppenstedt.

Six samples of liquors were sent out, consisting of two sapped oak liquors 1 and 1A., two sapped hemlock liquors 2 and 2A., and two oak-tail handler liquors 3 and 3A. The samples marked 1, 2 and 3 were regular sapped liquor samples, and those marked 1A., 2A., and 3A. were the same liquors, only with an additional amount of .25% of acetic and lactic acid (calculated as acetic acid on basis of A. liquors) added to them.

The instructions sent out were to compare the following six

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THE DETERMINATION OF SOLUBLE SOLIDS.

By H. C. Reed.
(Chairman 1906 Sub-Committee.)

The following instructions were sent to the members of the Committee:—

Soluble Solids Determinations.

Dilutions:—

Chestnut Extract	16	gms. per litre
Hemlock "	14	" " "
Solid Quebracho Extract	6	" " "

STUDY I.

(a). Determinations on three samples of extracts with above dilutions, according to the present official method. State temperature during filtration and time taken for entire filtration of the 250 cc. Also state whether filtrate is clear, brilliant, or cloudy.

(b). Determinations with the above dilutions, according to the present experimental method, as follows: "Pipette 100 cc. for total solids at room temperature.

Stir up 75 cc. solution with one gram kaolin, pour upon single pleated filter paper, No. 590, S. & S., 15 cm., filter, and return filtrate for one hour, keeping filter full.

Have cooled a sufficient portion of the original solution to 20° C., and after removing solution from the filter by decantation, proceed with filtration of cooled solution, discarding at least 10 cc. of the filtrate and collecting the first 100 cc., thereafter coming clear. Allow filtrate to come to room temperature before pipetting."

State temperature at conclusion of filtration and time taken for filtration of the 100 cc. used for soluble solids. Also whether filtrate is clear, brilliant or cloudy.

STUDY II.

Soluble Solids determinations with three samples of extract, using dilutions that will give solutions standing 10° Barkometer and proceeding according to Study I *(b)*. For total and soluble solids, pipette such quantity as will give approximately a weight of .7 gms for the total solids.

State the dilutions used and the number of cc. evaporated;

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time taken for filtration of the 100 cc. used for soluble solids and appearance of solution, whether clear, brilliant or cloudy.

STUDY III.

Soluble solids determination by means of the centrifuge.

To ascertain whether under similar conditions of manipulation the centrifuge separates out a constant amount of residue.

Conditions of temperature to be similar to that of Study I *(b)*. Clarity of soluble solids solution to be noted and time taken.

NOTE.—The Chairman has enclosed a blank table to be filled out with the statistics required, and requests that care be observed that none of the items be omitted.

Results were received from four of the five members of the Committee appointed and these are included in the following tables:—

STUDY I.

Official Method.

CHESTNUT EXTRACT (a)

	Dilution gms. per L.	Total Solids	Soluble Solids	Insolubles	Appearance	Time Min.	Tempt. Deg. C.
G. D. McLaughlin	16.0292	39.62	38.89	.73	Brilliant	80	24
J. V. R. Evans....	16.0	38.68	38.37	.31	Clear	90	21
M. F. Nichols.....	16.0	38.68	38.50	.18	Brilliant	90	22
H. C. Reed (1)....	16.0	38.91	38.73	.18	"	50	21.5
" (2)....	16.0	38.91	38.73	.18	"	43	23.5
Average		38.96	38.64	.32		71	22.4
Maximum		39.62	38.89	.73		90	24
Minimum		38.68	38.37	.18		43	21
Greatest Difference		.94	.52	.55		47	3

Experimental Method.

CHESTNUT EXTRACT (b)

	Dil. gms. per L.	T. S.	S. S.	Ins.	Appearance	Time Min.	Tempt. Deg. C.
G. D. M.	16.1090	39.55	38.98	.57	Brilliant	60	25
J. V. R. E.	16.0	38.68	38.12	.56	Clear	105	21
M. F. N.	16.0	38.68	38.62	.06	Brilliant	30	22
H. C. R. (1).....	16.0	38.91	38.82	.09	"	24	21
H. C. R. (2).....	16.0	38.91	38.75	.16	"	22	21.5
Average		38.95	38.66	.29		48	22.1
Maximum		39.55	38.98	.57		105	25.0
Minimum		38.68	38.12	.06		22	21.0
Greatest Difference		.87	.86	.51		83	4.0

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Official Method.

QUEBRACHO EXTRACT (a)

	Dil. gms. per L.	T. S.	S. S.	Ins.	Appearance	Time Min.	Tempt. Deg. C.
G. D. M.	6.0030	89.55	81.15	8.40	Clear	140	24
J. V. R. E.	6.0	87.03	78.75	8.28	Cloudy	210	19
M. F. N.	6.0	88.83	80.16	8.67	Clear	90	23
H. C. R. (1)	6.0	88.68	80.08	8.60	"	245	21
H. C. R. (2)	6.0	88.49	80.47	8.02	Cloudy	206	22
Average		88.52	80.12	8.40		178	21.8
Maximum		89.55	81.15	8.67		245	24.0
Minimum		87.03	78.75	8.02		90	19.0
Greatest Difference		2.52	2.40	.65		155	5.0

Experimental Method.

QUEBRACHO EXTRACT (b)

	Dil. gms. per L.	T. S.	S. S.	Ins.	Appearance	Time Min.	Tempt. Deg. C.
G. D. M.	6.0016	89.15	80.77	8.38	Clear	97	25
J. V. R. E.	6.0	87.03	79.55	7.48	Brilliant	150	23
M. F. N.	6.0	88.83	78.83	10.00	Clear	60	23
H. C. R. (1)	6.0	88.68	80.06	8.62	"	103	22
H. C. R. (2)	6.0	88.49	79.93	8.56	Brilliant	109	21
Average		88.44	79.83	8.61		104	22.8
Maximum		89.15	80.77	10.00		150	25
Minimum		87.03	78.83	7.48		60	21
Greatest Difference		2.12	1.94	2.52		90	4

Official Method.

HEMLOCK EXTRACT (a).

	Dil. gms. per L.	T. S.	S. S.	Ins.	Appearance.	Time Min.	Tempt. Deg. C.
G. D. M	14.1590	49.93	45.14	4.79	Cloudy	250	24
J. V. R. E	14.0	48.95	45.67	3.28	"	210	19
M. F. N	14.0	49.56	45.64	3.92	Clear	155	23
H. C. R. (1)	14.0	49.71	46.11	3.60	Cloudy	350	21.5
H. C. R. (2)	14.0	49.81	46.61	3.20	"	195	22
Average		49.59	45.83	3.76		232	21.9
Maximum		49.93	46.61	4.79		350	24.0
Minimum		48.95	45.14	3.20		155	21.5
Greatest Difference		.98	1.47	1.59		195	2.5

Experimental Method.

HEMLOCK EXTRACT (b).

	Dil. gms. per L.	T. S.	S. S.	Ins.	Appearance.	Time Min.	Tempt. Deg. C.
G. D. M	14.0920	50.17	44.77	5.40	Cloudy	345	25
J. V. R. E	14.0	48.95	45.31	3.64	"	210	23
M. F. N	14.0	49.56	44.50	5.06	Clear	105	23
H. C. R. (1)	14.0	49.71	45.19	4.52	"	160	22
H. C. R. (2)	14.0	49.81	45.29	4.52	"	161	22
Average		49.64	45.01	4.63		196	23
Maximum		50.17	45.31	5.40		345	25
Minimum		48.95	44.50	3.64		105	22
Greatest Difference		1.22	.81	1.76		240	3

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REMARKS UPON STUDY I.

As the results show, less concordance was obtained by the time-contact method than by the present official method. The Chairman feels that this may be in part due to lack of acquaintance with the manipulation of the time-contact method. The average insoluble in the case of the Chestnut extract was lower by the time-contact than by the official, but the opposite was the case with the Hemlock and Quebracho extracts. However, it is to be noted in this connection, that the appearance of the filtrates with the last two extracts mentioned was not so clear by the official method, and the results are more probably nearer correct by the time-contact.

Attention is called to the results obtained by the Chairman on the Quebracho and Hemlocks in analyses (1) and (2). These analyses were made some days apart and the concordance in the insolubles figure by the time-contact will be noted by comparison with the lack of agreement by the official method.

The Hemlock extract would not give a clear filtrate by the official method and in analysis (2) of the Quebracho extract the filtrate was distinctly cloudy. In opposition to this, filtrates from the time-contact method were in every instance clear, and the Chairman feels assured that a better acquaintance with the method will demonstrate that this is almost invariably the case with such extracts.

The Chairman does not feel assured that the results of the Committee's work are at all conclusive, and recommends that the time-contact method be given a trial in the general collaboration.

STUDY II.

Experimental Method on 10° Liquor.

CHESTNUT EXTRACT

	Dil. gms. per L.	T. S.	S. S.	Ins.	Appearance	Time Min.	Tempt. Deg. C.
G. D. M.	52.1613	39.35	38.74	.61	Brilliant	180	26
J. V. R. E.	60.0	38.56	38.35	.21	Clear	45	22
M. F. N.	71.60	37.93	37.70	.23	Brilliant	105	23
H. C. R. (1)	70.0	38.77	38.13	.64	"		
H. C. R. (2)	70.0	39.04	38.18	.86	"		
Average		38.73	38.22	.51			
Maximum		39.35	38.74	.86			
Minimum		37.93	37.70	.21			
Greatest Difference		1.42	1.04	.65			

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Experimental Method on 10° Liquor.

QUEBRACHO EXTRACT

	Dil. gms. per L.	T. S.	S. S.	Ins.	Appearance	Time Min.	Tempt. Deg. C.
G. D. M.	30.0016	88.98	80.06	8.92	Clear	180	26
J. V. R. E.	29.0	86.58	72.12	14.46	Cloudy	105	22
M. F. N.	29.0	86.85	71.29	15.56	Clear	Over Night	23
H. C. R. (1)	31.75	87.67	72.38	15.29	Brilliant		
H. C. R. (2)	31.75	88.49	72.74	15.75	"		
Average		87.71	73.72	13.99			
Maximum		88.98	80.06	15.75			
Minimum		86.58	71.29	8.92			
Greatest Difference		2.40	8.77	6.83			

Experimental Method on 10° Liquor.

HEMLOCK EXTRACT.

	Dil. gms. per L.	T. S.	S. S.	Ins.	Appearance.	Time Min.	Tempt. Deg. C.
G. D. M.	45.2377	50.26	43.99	6.27	Cloudy	480	26
J. V. R. E.	50.0	48.51	41.94	6.57	Brilliant	135	22
M. F. N.	57.34	48.74	38.30	10.44	Clear	Over Night	23
H. C. R. (1)	56.0	49.51	41.74	7.77	Brilliant		
H. C. R. (2)	56.0	49.44	41.91	7.53	"		
Average		49.29	41.58	7.71			
Maximum		50.26	43.99	10.44			
Minimum		48.51	38.30	6.27			
Greatest Difference		1.75	5.69	4.17			

Remarks Upon Study II.

It is merely necessary to refer to the tables to demonstrate that this method, so far as the concordance of the results obtained goes, is inferior in every respect to our present method and the Chairman would recommend that it be given no further consideration.

Personally, the Chairman is of the opinion that since it is admitted that analyses at different dilutions will give different results, it is far preferable to use a dilution that is capable of returning figures in concordance rather than to adopt a dilution that will give results similar to those obtained under Study II.

STUDY III.

No work was done by any of the members of the Committee with the centrifuge. This method has been tested by members of the I. A. L. T. C., who agree that it will not give clear soluble solids solutions. This is also the case with the filter-candle method, which is now being criticised by members of the International Association for the very reason mentioned, *viz:* failure to give clear filtrates. (*Collegium*, 1906, *No.* 209).

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In concluding, the Chairman would like to call attention to the uneven character of the 590 S. & S., 15 cm., paper, which has been furnished during the past year. He has been obliged himself to return package after package of the paper, mainly because it not only utterly refused to give clear filtrates, but frequently would not bear the weight of the solution.

JUNE MEETING OF THE BRITISH SECTION.

I. A. L. T. C.

At the meeting of the British Section on June 16th, Dr. Parker pointed out to members that the German Section had adopted the use of lightly chromed hide-powder as the official hide-powder for the German Section. The Chairman pointed out that the German Section was within their rights under Rule 4, provided they stated that chromed hide-powder was used on their official reports. Mr. Kopecky proposed, on behalf of Prof. Procter, the following resolution, which was seconded by Mr. A. Seymour-Jones, and carried unanimously: "The British Section regrets that the German Section did not await the decision of the Executive Committee before taking action on the subject of hide-powder."

A further resolution was also proposed on behalf of Prof. Procter by Mr. Kopecky and seconded by Mr. Charles E. Parker: "The British Section, while anxious loyally to adhere to all prescriptions of the Association as to quality of material employed, methods of manipulation, and, where necessary, forms of apparatus, strongly depreciate and will consistently oppose, all limitations to individual firms, manufacturing articles for profit or by private methods." This resolution was supported by Messrs. O'Brien, A. Seymour-Jones and Dr. Parker, and carried unanimously.

Mr. Kopecky proposed, on behalf of Prof. Procter, that the British Section should propose the decision of the resolution of the Conference of Turin demanding the filtration of all tanning solutions through the candle filter; and directing that all matters that passed through under normal conditions be regarded as soluble, since it can be shown that candles, as at present supplied,

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pass certain extract solutions quite turbid and containing sometimes 8% to 10% of matter which can be removed by clear filtration. They propose, in place of this rule, to return to that previously in force, which permits the use of the candle or any other effective mode of filtration and to define a clear filtrate as one which is free from visible opalescence, since such filtration can always be obtained by the use of No. 605 paper and kaolin with an approximate correction for absorption. This resolution was seconded by Mr. A. Seymour-Jones. After considerable discussion, Dr. Parker proposed the following amendment: That in cases where the filter candle does not filter clear the first time the solution should be passed through the candle a second time or until clear. If necessary, kaolin may be added to assist in obtaining a clear filtrate. This was seconded by Mr. O'Brien. The amendment was carried, only two voting against it.

Mr. Kopecky, on behalf of Prof. Procter, then proposed that the British Section approve the suggestion of Prof. Procter that all matters absorbed by hide-powder which are soluble or will pass through the candle or other effective filter at 100° C. should be reckoned as tanning matters, since it can be shown that even those insoluble at 15° C. are capable of producing leather, and that when desired the proportion of soluble matter and insoluble matter at 15° should be determined as at present and returned respectively as easily and difficultly soluble tanning matters. This proposition was seconded by Mr. A. Seymour-Jones, and raised considerable discussion. Dr. Parker pointed out that this was liable to be very misleading. Mr. Charles Parker agreed that under certain circumstances useful information might be obtained. It was agreed that the matter was extremely interesting. Those at the meeting were of the opinion that the resolution should not go forward to the Conference as a resolution of the British Section. Mr. Charles Parker proposed as an amendment, that the British Section desire to bring this matter forward as a suggestion for the next Conference, and that the matter be discussed there, and that if desirable, a commission should be appointed to investigate the subject; this was carried.

Mr. Kopecky gave a demonstration of the chromed hide-powder process of analysis, and communicated a mass of figures, tending to prove that concordant results were obtained by using the chromed hide-powder obtained from chromed hide-shavings

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after suitable washing and grinding, and showed that more concordant results were obtained than have been obtained by any other chromed hide-powders. This view was borne out by Dr. Parker, Mr. Wood, and Dr. Turnbull, who had sent in some results to Mr. Kopecky. After considerable discussion, the following resolution was proposed on behalf of Prof. Procter, by Mr. Charles E. Parker: "That the British Section, while fully recognizing the services of the hide-powder filter method in times past, is of the opinion that it should now be abandoned for a more scientific method in which a definite quantity of hide-powder rendered fully insoluble by chroming is fully brought in contact with a definite quantity of tanning solution by maceration, or shaking. It approves in principle the method outlined by F. Kopecky, and recommends the appointment of a small commission, including representatives of the American Leather Chemists' Association and of the K. K. Research Station in Vienna, to consider and, if possible, to formulate a really international method, and to report to the Executive Committee of the I. A. L. T. C., which should have full power to authorize its adoption." This was seconded by Mr. A. Seymour-Jones and passed unanimously. The meeting congratulated Mr. Kopecky on his valuable work, and thanked him for communicating the large number of figures and results he had obtained.

LIME SPOTS ON COLORED LEATHER.

(W. Eitner, in "Der Gerber.")

The lime spots on colored leather are caused when the hides, after dehairing, are placed in too hard water (bicarbonate). These spots can be prevented, by removing the carbonate of lime, precipitated in the grain, with acid (not sulfuric and oxalic acid). If the lime salt remains in the grain, the light lime spots will appear. The simplest way to remove the spots, in leathers to be dyed, is to place the leather, after being softened and washed, in a 2% solution of hydrochloric acid for two hours, keeping it agitated. It is then thoroughly washed.

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PATENTS.

U. S. Pat. 825,852, Issued July 10, 1906, to J. L. Martin, Terrell, Texas.

Process of Tanning.

A composition for tanning is made of ten gallons broom weed ooze; four pounds gum gambier; one pound alum; two pounds common salt; three pounds persimmon bark, or in the above proportions. The broom weed ooze is made by boiling thirty pounds of broom weed in fifteen gallons of water for from one to two hours, or until volume is reduced one-third. Gambier is dissolved and added to the ooze, and the alum and salt are then added and dissolved. Freshly peeled persimmon bark is added when the hides are put in the ooze.

The hides are soaked, fleshed, limed, dehaired, and are then bated with water; after coloring, the hides are placed in the ooze which is claimed to tan a light hide in six hours and the heaviest hide in ten days.

U. S. Pat. 825,808, Issued July 10, 1906, to Alexander Classen, Aachen, Germany.

Process of Converting Cellulose of Wood into Fermentable Sugars.

The inventor refers to his earlier patent covering his process of converting cellulose into sugar by treating comminuted wood with sulphuric and sulphurous acids under heat and pressure. This process has been practically effected only on treating pine or other woods containing little or no tannic acid. The patent now issued describes treatment of tannin-bearing woods, such as chestnut, oak, etc., to increase the yield of fermentable sugars when these latter woods are subjected to the original process of the inventor.

The inventor finds that the smaller yield of alcohol from tannin woods is due to the fact that the gallic acid formed by the transformation of tannic acid exercises an injurious action upon the fermentation of the sugar solution. By removing this gallic acid from the sugar solution obtained from tannin woods, the fermentability is increased so that the alcohol yield is substantially the same as from pine.

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The inventor proposes to remove the gallic acid by precipitating it as an insoluble compound, by combining it with a metal. A salt of iron, e.g. ferric sulphate, is added to the sugar solution, and after this the free acid is neutralized with calcium carbonate, and then pulverized calcium hydroxide is added in small portions until solution is slightly alkaline. The precipitate formed contains in addition to the calcium sulphate and iron gallate, some ferric hydroxide, and may, owing to the latter, be employed for treating fresh tannic solutions.

U. S. Patent 825,435, Issued July 10, 1906, to Carl Weilbier, Hanover, Germany.

Impregnated Gloves.

The inventor impregnates chamois, or other glove leather, with a chemical solution which insulates the glove and protects the wearer from electric currents. The skins are degreased by boiling in soda, and then boiled in a 25% solution of the compound, rinsed out in water and dried. The compound consists of iron sulphate, sodium nitrate, sulphuric acid and water, each in specified amounts.

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Journal of the

American Leather Chemists Association

Vol. I. SEPTEMBER, 1906 No. 9

W. H. TEAS, ... Editor
L. E. LEVI,
A. W. HOPPENSTEDT, } . . . . . Associates
R. P. CUSHING,
H. C. REED, ... Business Manager

Correspondence pertaining to the Editorial Department should be addressed to the Editor at Ridgway, Pa.

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The American Leather Chemists Association

G. A. KERR, W. H. TEAS. . . . . Past Presidents

OFFICERS, 1905-1906

PRESIDENT—H. C. Reed, Stamford Conn.

VICE-PRESIDENT—C. W. Norris, Milwaukee, Wis.

SECRETARY-TREASURER—W. K. Alsop, New York City.

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EDITORIAL.

At a recent meeting of the German Section of the I. A. L. T. C., the German extract manufacturers mildly remonstrated against the repeated changes in the official method of tannin analysis. Our own Association might wisely forestall a similar complaint from commercial interests, by providing for the continuance of the present method of extract analysis during a considerable term. Frequent changes in the method beget doubt in the mind of the layman as to our confidence in the method; and these changes are

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annoying and confusing to the operative who has probably just become accustomed to the provisions of a previous year. These remarks apply, of course, to minor changes in the method which cause changes in results so small as to be accounted for by the personal error. Radical changes in the method should be perfected *before* incorporation in the method, thus preventing frequent changes after the adoption of a radical provision which has not been perfected.

Methods for the determination of tannic acid, as tannic acid, are undoubtedly valuable; but in the commercial tanning world, these must give place to a less scientific and more practical method along the lines of actual tannery operations, giving comparative results and which tends to indicate the amount of the tanning agent which will combine with the hide, rather than the percentage of any specific tannin. This being true, the ideal method would involve analysis with actual tanning strengths of solutions, at actual tanning temperatures, and the use of hide under tannery conditions. In a general way, our method is patterned after tannery operations; in some particulars our method can never approach the ideal; in other particulars, there are of us some sanguine bodies whose hopes may some day be realized. Under these circumstances, that method is best which in a general way follows the tannery the closest, duplicating conditions when practicable, deviating only so far to insure concordance and sacrificing tannery conditions only in the interests of concordance.

The A. L. C. A. method calls for an analysis temperature at which there is least variation of insolubles, and which at the same time is very close to the average tannery temperature. We slightly chrome our hide powder to elude soluble hide substance; a more complete chroming might be claimed to be more desirable, but this is a further deviation than necessary. Our method prescribes a weaker dilution than tannery practice, simply because as yet we have found no quick method of filtering which gives consistent results on strong liquors.

The method, as it now stands, gives comparative results in the hands of different operators, and is the method that suffers least in the hands of different operators. If its provisions are strictly adhered to, concordant results will follow. If its provisions are not carried out—why blame the *method?* We do not claim that

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it gives actual tannic acid values, but it has established standards and the merchant and the practical tanner understanding the relations of the standards to their operations, can rightly interpret the comparative values fixed by subsequent analyses by this method.

Practice and familiarity will improve concordance, and we trust that the Association will wisely refrain from present amendment and allow the method to be tested by time.

****

The work of our Association in the past has been mainly along the lines of extract analysis, but we are now engaged in standardizing methods of analysis for the other determinations making up the routine and occasional work of a leather laboratory. The experience gained in developing an extract method will undoubtedly stand us in good stead in this work, and yet there are conditions to be met with which were unknown in the extract problem.

These new official methods should have the utility of our extract method; they should admit of every day use, and should not be glass-case methods to be laid on the shelf and used only for collaborative work, or on demand for special referee occasions. Such methods, albeit they are more scientific and may possibly result in a few hundredths percent closer to the supposed theoretical, defeat the purpose of a truly official method. They take more time and attention, and this precludes the possibility of the average leather chemist becoming familiar and proficient with them. As yet, no one can say authoritively which of two tannin results agreeing within the range of personal error is correct; proof by a different manner of analysis is necessarily barred. The utilitarian idea must obtain in this work, so let us have official methods that will give comparative results, and that all will use in their daily work.

****

The 1906 Conference of the I. A. L. T. meets at Frankfurt a/M Germany, on Sept. 10th. Messrs. H. C. Reed, C. Eachus and E. C. Klipstein, of this Association, will attend the Sessions of the Conference. Messrs. Reed and Eachus are the accredited delegates from the A. L. C. A.

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SOLE LEATHER BLEACHING.

By Simon C. Hemie.

The term, "bleaching," as applied to the usual process of brightening side or sole leather, is a misnomer. Real bleaching agents are occasionally used, but the most general method of "bleaching" consists in dissolving the mechanically held tannins from the exterior of the leather by means of an alkaline solution, neutralizing and washing off the alkali remaining on the leather, and finally washing off the excess of acid used to neutralize the alkali.

From three to six separate baths are used; the alkali, acid and water baths are essential; the use of more than these, however, often results in brighter leather and greater economy. If one or even two baths of water are given the leather before immersing in the alkali, considerable surface tannin is removed, and as this is not contaminated with soda or acid it may be saved and used in the yard or leach-house. Where water baths are used, a weaker soda solution may be used. After the soda bath, one or two acid baths may be used; if two are used, the second is only a weak acid used as a precautionary measure to neutralize any soda which may have escaped the action of the first acid bath. The final bath is of water and in some tanneries two waters are used after the acid; barium chloride being occasionally used in the last bath to precipitate any remaining traces of acid.

The vats containing the bleaching solutions are placed in a row, the leather hung on sticks going in the first vat and remaining the desired period, is then raised, drained and then immersed in the second bath, and so on through the series.

Mechanical appliances for handling and moving the leather vary according to the ideas of the tanner; they range from the primitive and back-breaking hand-dipping, to the latest hydraulic apparatus which lifts the sides from all vats at once, moving each lot to the next bath, loading a new lot of unbleached leather and dumping the bleached sides.

The alkali most commonly used is soda carbonate, either as sal-soda or in the concentrated forms of soda-ash. Combinations of

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caustic soda and soda-ash are also used, an alkali recently marketted for this purpose consisting of soda-ash with about 10% added caustic soda. Cost is the only factor determining the form of alkali used for sole leather bleaching; the action is purely solvent, and in spite of claims as to "penetration," the cheapest unit of alkali is the one to use. Because of its low cost, sulphuric acid is generally used in the acid bath.

The baths are kept from 100° F. to 120° F. temperature, the temperature being quite an important item in getting good results. The soda and acid baths are made up with the requisite amounts of each, the baths being well plunged before use; both soda and acid baths are strengthened after bleaching a certain number of sides during the day. The amount of soda used depends on the character of the stock, length of time in the soda bath, and on the color desired; the amount of acid should, of course, be regulated by the quantity of soda. Not more than 100 lbs. of 66° Be. acid should be used for every 250 lbs. of sal-soda used, and where there are two water baths after the acid, less than this proportion may be used.

Controlling the strengths of the soda and acid baths is sometimes a difficult matter, particularly when the stock is not running uniformly. Once the proper amounts are determined, the baths will run for a considerable time on uniform stock, using the set schedule of soda and acid. Laboratory control of this work is occasionally required, and while simple titration of a diluted sample from the acid bath is generally possible, the soda bath is too highly colored to admit of this treatment. It is best to evaporate 50 cc. in a platinum dish to dryness, and char at a low red heat; break up the residue, treat with hot water three or four times and then titrate with standard acid and methyl orange.

It is frequently desirable to know the amount of material removed from the leather by the bleach, and the following method gives fairly accurate figures:—

Take samples of all baths before starting the day's work, and samples of all vats at the close of the day. Evaporate and dry 50 cc. of each sample, weigh, and subtract the combined "morning" percentages from the combined "evening" percentages. Multiply this difference in percentages by the weight of water in a single vat, and from this result subtract the weights of soda

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and acid added during the day. (Acid weights should be taken at 100% and sal-soda weights at 40% actual solids). The remainder will be the approximate amount of organic material removed in all the vats, and this figure divided by the number of sides bleached will give the amount of solids removed per side.

If one were absolutely certain that the leather going in the soda vat was uniform as to size and condition, the effects of different alkalies might be studied with profit. A method for determining the amount of tanning material removed by the soda vat, is to evaporate and dry 50 cc. of the liquor in a platinum dish; weigh and then char the contents and determine the alkali in the charred mass by titration. The difference between the weight of the dried residue, and the weight of the alkali (figured in its form at the drying temperature), will give the organic matter in 50 cc., and knowing the size of the vat and the number of sides bleached, the amount removed per side may be readily figured.

EXTRACTORS FOR TANNING MATERIALS.

By W. H. Teas.

The too frequent wide variation in analytical results on new and spent tanning materials, is more often due to improper sampling, poor preparation, and incomplete extraction than to the method of analyzing the extracted matter. In this work, the effect of imperfections of the method and of the operator has been insignificant as compared with the influence of sampling, preparation and extraction.

As a rule, the chemist is not responsible for the sampling; and if his directions for sampling are not carried out, he cannot be blamed for having an unfair sample to analyze.

Now that the degree of preparation has been definitely prescribed in our Official Method, chemists following the method cannot ascribe variation in results to this cause.

The chemist is always responsible for the extraction, and if he makes an incomplete extraction of a low average sample, his results will be far from correct, and in many instances will cause wrong deductions by those interested, and possibly financial loss.

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During the past two years the subject of extraction has been closely studied by many American leather chemists, and several different types of extractors have been introduced and advocated; all of the tannin extractors thus far devised may be divided into three general classes; they extract, either, 1st. By a simple percolation, the extractives being carried away from the apparatus. 2nd. By a cumulative percolation, the extractions remaining in the apparatus, the solvent being alternately volatilized and condensed. 3rd. By a combination of the operations outlined for the first two classes.

The extractors coming under the first classification are *essential* for the extraction of *some* tanning agents, and *can* be used on *all* unbleached materials, but unless the material is very rich in tannin (allowing of small charges), ultimate extraction is reached only after a long period of time, and results in a large amount of weak extractive solution. The extraction of spent tanning materials by this class of extractions is not practicable.

Extractors of the second class have been most popular for spent materials, and for unleached materials not harmed by an initial boiling water extraction temperature.

The third class of extractors are fast being recognized as the most reliable, and, of course, can be used on both new and spent tanning materials. The possibility of different ways of operation, makes them the most valuable extractors for general laboratory work; and except for some uncommon tanning materials, they are practically universal extractors.

Dr. Procter's extractor is the pioneer and probably the best known of extractors in the first classification, but as ordinarily used it is difficult to regulate the temperature and flow of the water, and the apparatus is not sufficiently permanent for general routine work. As the result of experimenting to produce a more permanent and practical extractor of this class, I designed the percolator extractor exhibited and explained at Washington last November. Since then, I have grouped single units in a common jacket, as illustrated in Fig. I., and effected a saving in time necessary to regulate space, and of heat required to operate. Except for the grouping the extractor is the same as the original single unit type; the jacket (A) being fed through pipe (B) from a reservoir at a sufficient height to raise the boiling point of the

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water. Heat is applied to the bottom of the extractor, and water regulated by the cocks (C) falls on the material in the cylinders, and after percolating, is delivered as desired through either of the cocks (D).

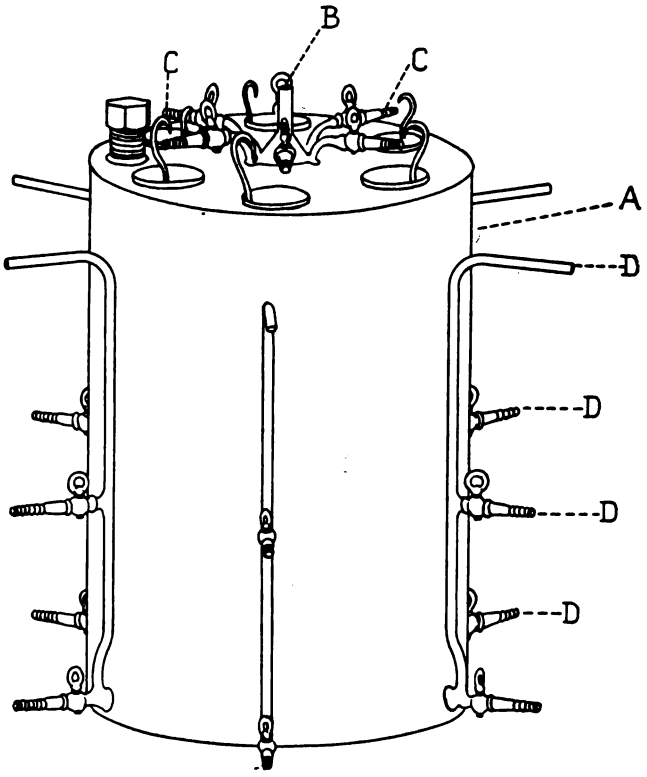

Fig. 1.

The cylinders are 2 inches inside diameter, as experience has indicated that this size gives better results than the narrower cylinders used in the early form of this extractor. Theoretically, the best results should be obtained in a narrower column, but with the present official preparation requirements, and the high temperature maintained at the latter part of the extraction, a narrower column gives no better extraction and causes trouble from packing, channelling and spurting. This extractor does excellent work on Sumac, Mangrove, Myrabolams, Algorabilla, etc., and will extract any *new* material, but is subject to the previously

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stated objections to the percolator type of extractor when working on Oak and Hemlock Barks, Chestnut Wood, or on any spent tanning material.

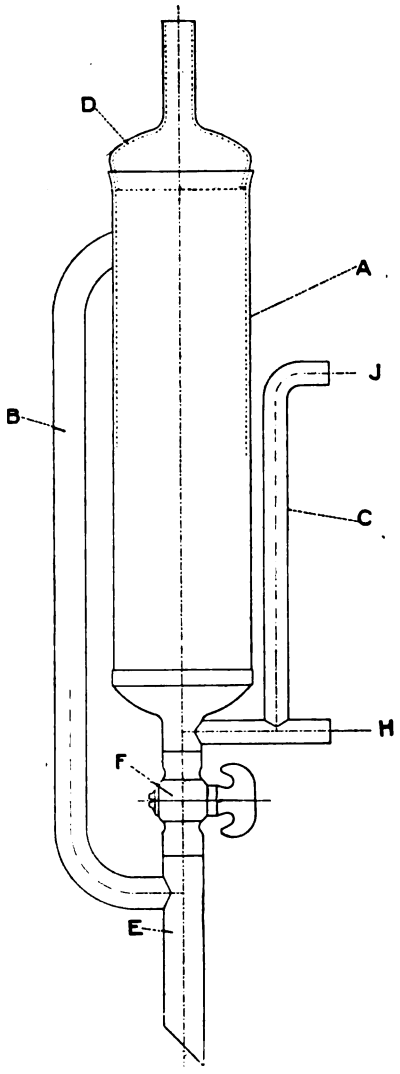

Fig. 2.

The Soxhlet extractor is the best known among extractors of the second class, and under the best working conditions it will ex-

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tract as well as any known extractor. Objections to this class of extractors because of the initial high temperature of extraction, the continuous boiling of the extractives, and also, in the case of the Soxhlet, because of the frequent clogging of the siphon, have developed the third and newest class of extractors which combine the meritorious features of the other two classes.

In the third class belongs an extractor with which our own laboratories are now being equipped. The Zukolwski side tube extractor has been suggested and advocated for use in tannin work, and to this I have added means of drawing off the stronger portions of the extractives, and of instantly changing the apparatus from a percolator extractor to a cumulative condensing extractor and *vice versa.*

The new style of extractor shown in Fig. 2 consists of a shell (A) with side tube vapor conveyor (B), delivery tube (C), cap (D), mounted on a stem (E) with cock (F). The stem (E) is connected with a reservoir by either a perforated rubber stopper, or brass plug with ground joint, and the cap (D) is connected with a water jacketted condensing tube.

In extracting materials unharmed by a high initial extraction temperature, the material is brushed in on the perforated porcelain disk covered with a thin layer of cotton or a double filter paper, and a second perforated porcelain disk covers the material. After connecting with reservoir and condenser, and applying heat to reservoir, the cock (F) is closed and either upper (J) or lower (H) rubber tube on the delivery tube are opened by removing a pinch-cock. The strong extractives are delivered through the opening in the delivery tube, and when the desired amount has come over, the opening in the delivery tube is closed, the cock (F) opened and a cumulative extraction continued as long as desired. Progress of extraction may be noted by closing main cock (F) for a moment, opening lower side cock (H) and collecting and testing some of the percolating liquor. When extraction is complete, the strong and weak solutions should be well mixed while hot, and allowed to cool to analysis temperature. Our experiments have shown that in extracting this class of materials, it is immaterial whether the strong extractives come through the upper or lower openings in the delivery tube, or in other words: whether the material is flooded, or subjected to a true percolation.

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However, it is well to shut *all* cocks for a few minutes just before opening the cock (F), so that the material will be fully flooded at least once, and also to remove any solubles that may escape the percolation.

This extractor may be used on materials affected by a high initial temperature, by closing cock (F), charging and flooding with cold water to just below the level of the upper opening (J) in the delivery tube; the strong portions are then removed through the upper opening (J), the hot water falling from the condenser being cooled as it gradually works through the material, and the latter is subjected to a gradually increasing extraction temperature. The extraction of these materials should proceed rather more slowly during the early part of the process than in the later.

Where the extraction is conducted during the day, the extractors may be charged and flooded with warm water the previous afternoon, and the soaking the material receives during the night will materially lessen the required time for complete extraction.

The maximum time for complete extraction with this apparatus working under favorable conditions is ten hours. In some cases there may be a small amount of extractives remaining in the material, but the tannin remaining will not be appreciable. By "favorable conditions," I mean that the material must be ground to pass a twenty-mesh sieve, the condenser tube must be at least ½-inch inside diameter, and must have a cold water jacket at least 12 inches long; and (after strong extractives have been removed) the reservoir must supply sufficient steam so that "rattling" in the condenser tube is imminent.

When the material has been soaked over night in the extractor, the extracting time in most cases may be reduced to eight hours.

NOTES.

CURIOSITIES OF CUTCH.

(From Journal of the Society of Arts.)

Amongst the many curious and complex questions which come before the Law Courts, was one, which some time since, occupied the attention of the Court of Appeal, and which will have a pecu-

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liar interest to those whose tastes lead them to the consideration of plant products, and more especially to such as have also a leaning towards antiquities.

To explain, it will be necessary to quote the facts of the case as reported in the daily press. The question was as to the validity of a custom said to have prevailed in the parish of Walmer, in Kent, for the inhabitants, being fishermen, to spread their nets to dry on a piece of ground, called the "beach ground," of about eleven acres in extent, covered with shingle, near the sea. The fishermen claimed this right by virtue of an immemorial custom, and the application was for an injunction to restrain the owner of the ground from building upon it.

The part of the case in which the interest chiefly lies, is described in the following notes from the legal report. It seems that the fishermen down to twenty-five or thirty-five years ago, used to tan or cutch (*i. e.*, preserve by means of an astringent plant imported from the Malay Archipelago), their nets immediately before the commencement of the mackerel and herring fisheries, and dried them on the beach ground. At the period named, the practice of tanning or cutching nets was discontinued, and instead of it the nets were oiled and dried on the beach. The nets take longer to dry when oiled than when tanned or cutched. The change was probably due to cotton being used instead of flax or hemp as the material of which the nets are made. The claims of the fishermen were disputed on two grounds: First, although it was admitted that a custom for fishermen to dry nets that had become wetted by the sea in fishing, was good, it was contended that there could not be a valid custom to dry nets wetted by tanning, cutching, or oiling, inasmuch as these are comparatively modern processes, and a custom must date from the beginning of the reign of Richard I., A.D., 1189. This contention was over-ruled, the Judge remarking that the ground upon which a custom to dry nets in the strict sense had been upheld was, that it was in favor of commerce and navigation. Tanning, cutching, or oiling nets tended to preserve them and to make them useful for a longer period, and the fishermen ought not to be deprived of the custom simply because they had taken advantage of modern inventions or new operations which did not throw an unreasonable burden on the land-owner. The second objection was that in consequence

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of the receding of the sea in the neighborhood during the last fifty or sixty years, and that as custom is a local law, which must have existed from time immemorial, it cannot be applicable to land which can be shown to have emerged from the sea in modern times. This contention was also overruled on the ground that even if the sea had gradually and continuously receded, "that which cannot be perceived in its progress is taken to be as if it had never existed at all." In the view of the law, therefore, this was the same piece of land as that affected in the time of Richard I. The Walmer fishermen thus secured their right to dry their nets upon the beach ground, and an injunction was granted restraining the owner of the ground from building upon it.

All this, though very technical, is of considerable interest, when taken in connection with the history of cutch, or, as it is sometimes called, catechu. Cutch, as most people know, is the product of *Acacia Catechu,* a large leguminous tree belonging to that section of the order known as *Mimoseae.* It is common in most parts of India and Burma, extending westward to the Indus and eastward to Sikkim, and ascending to an altitude of 5,000 ft. The trees grow to a very large size, 70 ft. to 80 ft. in height, with a girth of from 8 ft. to 9 ft. being not uncommon. The tree is an extremely valuable one, whether for its timber, its gum, or for the more important product, cutch, which is far and away the most valuable of all. It is an astringent resinous substance obtained by boiling down chips of the heartwood until the decoction has become a thickened extract, which solidifies on drying and cooling. It is interesting, in connection with its use for tanning or "cutching" fishing nets by the Kentish fishermen, to know that the preparation of this extract dates from a very remote period. It is mentioned by Sanskrit writers, and in 1514, in a description of the East Indies, a drug is described, under the name of "cacho," which was exported from Cambay to Malacca, and was in all probability cutch. In 1574 an account of the plant and the process of preparing the extract was given by Garcia de Orta. In the seventeenth century the substance, under the name of catechu, began to attract attention in Europe. It came by way of Japan, being re-exported from that island, and was at first supposed to be a natural earth, to which the name of *Terra japonica* was given, a name which has since become attached to true catechu, pale catechu, or gambier, which is an extract prepared in a similar

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way from the leaves and young shoots of *Uncaria gambier,* a rubiaceous plant of the Malay Archipelago, and cultivated in Singapore and Sumatra. This is a drug which is used medicinally as an astringent, while black catechu, or true cutch, from *Acacia catechu,* is used chiefly for dyeing and tanning, and is identical with the substance used by the Kentish fishermen in preserving their nets from decay, and of which very large quantities are imported into Great Britain yearly.

* * * * * * *

A German contemporary gives the following qualitative test for the detection of cod liver oil, which is said to be recommended by Vogt:—Twenty drops of chloroform, forty of glacial acetic acid, and three drops of sulphuric acid are mixed in a test tube and allowed to cool, and shaken up with three drops of cod liver oil. A bright blue coloration is at once produced, changing in about twenty seconds to an olive green. It is pointed out that blubber oils, on the other hand, give a reddish-brown coloration, changing into a pale yellow-brown to a yellowish-green tinge.—*Leather Trades Review.*

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Journal of the

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Vol. I. OCTOBER, 1906 No. 10

W. H. TEAS, ... Editor
A. W. HOPPENSTEDT, } . . . . . Associates
R. P. CUSHING, }
H. C. REED, ... Business Manager

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ANNUAL MEETING

The annual meeting of the Association will be held in New York City, November 22, 23 and 24th, 1906.

ELECTIONS

The following persons have been elected to membership under date of September 7th, 1906:

ASSOCIATE.

Horton Johnson, Ridgway, Pa.

ACTIVE.

Charles R. Oberfell, 323 Academy St., Newark, N. J.

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The Secretary is pleased to call the attention of the Association to the following letter:—

INTERNATIONAL ASSOCIATION OF LEATHER TRADE CHEMISTS. (I. A. L. T. C.)

Herold's Institute, Drummond Road, Bermondsey,
London, S. E., Sept. 26, 1906.

British Section.

Mr. W. K. Alsop,
Hon. Secretary American Leather Chemists Association,
28 Ferry Street, New York.

DEAR SIR: At the recent Conference of the International Association of Leather Trades Chemists, held in Frankfort, a special resolution of thanks was passed to your Association for having sent over, as delegates, your President, Mr. Reed, and one of your leading members, Mr. Charles Eachus. It was desired by the Conference to convey to you this resolution, and to express to you the great pleasure it gave us to welcome your members, and also to express the hope that our two Associations may work in unison and concord for the benefit of the great leather trade, that we shall be able to interchange researches and ideas, and that it may be possible, through your Association, to form a closer bond of union between the members of both Associations, as many of the members of the I. A. L. T. C. are desirous of becoming foreign members of your Association.

Therefore, on behalf of the I. A. L. T. C., please express to all the members of your Association our sentiments of regard and esteem, and our hope that we may continue to work towards the same end, *viz.*, the elucidation of many of the complex problems connected with the leather trade.

Yours very truly,

Signed on behalf of the I. A. L. T. C.

DR. H. BECKER,
President.
DR. J. GORDON PARKER,
Hon. General Secretary.

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THE FRANKFORT a/m CONFERENCE OF THE I. A. L. T. C.

The Convention assembled on the morning of Monday, September 17th. Dr. Becker, the new President, was then installed in office and welcomed the members with an interesting Presidential address, which was followed by addresses by the Mayor and other officers of the City of Frankfort.

A commemorative medal was then presented to the family of the late Franz Kathreiner.

A report was then received from the Commission appointed to adjudge the award of the Klipstein Prizes, which reported in favor of awarding the prize upon the "Relative Value of the Hide Powder Filter Method and Shake Method of Tannin Analysis," to the essay submitted by Messrs. Paessler and Appelius, but regretted that the essays by Ferdinand Kopecky and H. C. Reed could not be considered, owing to the fact that neither had complied with the conditions imposed by the terms made in offering the prize. The second prize was not given, but the offer continued until the next meeting of the International Association of Leather Trades Chemists.

The Seymour-Jones Prize was then awarded Ferdinand Kopecky, for his work on the Hide Powder Question.

An interesting paper was then read by Dr. Lepetit upon the development of the Italian Leather and Extract Industry, whereupon the Convention adjourned to partake of a luncheon given by Herr Dreyfuss, Editor of *"Der Gerber."*

The Convention re-assembled in the afternoon and listened to the report of the Committees appointed to formulate a standard method for the analysis of Egg Yolk and for the analysis of Degras.

The Convention adjourned and re-assembled on the morning of the 18th. Prof. Procter immediately introduced a resolution to the effect that the filter-tube should be abandoned from January 1st, 1907, for the analysis of tanning materials and proceeded to support his resolution by pointing out the weakness of the filter-tube method through its excessive absorption of matters other than tannin in conjunction with its extreme uncertainty of yielding uniform results.

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Those taking part in the discussion that followed were Messrs. Prof. Parker, Eitner, Jean, Dr. Lepetit, Prof. Wislicenus, Dr. Turnbull, Dr. Becker, Dr. Paessler and Nihoul.

Dr. Paessler spoke in favor of his lightly chromed hide powder, used in the filter-tube and argued that with it, so many of the troubles experienced with the filter-tube method would be done away with.

H. C. Reed spoke on the many advantages possessed by the shake method.

The Convention adjourned without taking a vote upon the resolution.

Upon re-assembling in the afternoon, matters pertaining to the conduct of the Association were discussed and acted upon. Prof. Procter gave expression to the wish that between the International Association of Leather Trades Chemists and the American Leather Chemists' Association there be established a very friendly feeling, and that they get in touch with each other in their experiences and publications. The Convention agreed with this friendly proposal and authorized the Chair to take steps in this direction.

After further discussion pertaining to the welfare of the Association, the Convention adjourned for the day.

The Convention re-assembled on the morning of the 19th.

Herr Boegh delivered a short address, followed by Dr. Maschke and Prof. Procter. Dr. Baum then spoke upon a new electrical method for the estimation of tannin, followed by Mr. E. C. Klipstein, who drew attention to the great variation in results, particularly between chemists using the International method. Prof. Procter then withdrew his resolution to the effect that the filter-tube method should be abandoned after January 1st, 1907, and introduced in its place a resolution to the effect that the Conference should appoint a Commission to examine into the merits of the various methods of tannin analysis and report to the Executive Committee not later than April 1st, 1907. The Executive Committee to be empowered to make the method of analysis favorably reported upon by the Commission, the Official Method of the Association.

This resolution was adopted and the following members were appointed upon the Commission:—

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Prof. H. R. Procter, *Chairman;*
Dr. J. Gordon Parker,
Dr. George Klenk,
Valdemar Boegh,
Prof. Louis Meuinier,
Prof. Eitner,
Prof. Dr. Ed. Nihoul,
Doct. Giacinto Baldracco,
H. C. Reed.

The Commission appointed for the analysis of Degras made its report, which was adopted.

The Convention then adjourned to visit the Leather Manufactory of J. Mayer & Son and the Machine Manufactory of The Turner Company.

Upon re-assembling on the morning of the 20th, Mr. H. C. Reed spoke upon the line of work being pursued by The American Association, pointing out the advantages of the shake method over the International method, and discussing the new time contact method of soluble solids determination and pointing out the advantages of the combined evaporator and dryer, also the difficulty experienced in the extraction of crude tanning materials.

Dr. Paessler followed with a paper upon the detection of admixtures of tanning materials and gave an exhibit of color tests upon mordanted cloth, but with the conclusion that the method was not satisfactory for the purpose.

Discussion upon various subjects took place followed by papers by Mr. Kanschke upon recent experiences in leather dyeing and by Dr. H. Sickling upon different methods for the production of artificial leather.

After further general discussion, Mr. Eachus thanked the Association for their courtesy to the American representatives, and the Conference adjourned.

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THE RELATIVE VALUE OF THE HIDE POWDER FILTER AND THE AMERICAN SHAKE, OR CHROMED HIDE POWDER METHODS OF TANNIN ANALYSIS.

By H. C. Reed.

The two methods of tannin analysis of which this essay treats have for a number of years dominated all other methods as better expressing the actual tannin contents of tannin materials. The claims made for the merits of either method have been more or less fully put forward in the past. The members of the American Associations (the A. O. A. C. and A. L. C. A.) have been practically undivided in support of the Shake Method, but such has not been the case with the members of the International Association (the I. A. L. T. C.). Prof. Procter, the originator of the Filter Method, went so far at the Leeds Convention of the I. A. L. T. C., in 1902, as to offer a resolution calling for the adoption of the American Shake Method as the official method of the International Association, and the resolution failed by only a very narrow margin. However, at this same meeting, the following resolution was adopted: "(D) That the Filter Method of determination of non-tannins, as prescribed in the rules of this Association, remain the official method until the next conference, but that members be permitted to employ the chromed hide powder method of the conference of the American Association of Official Agricultural Chemists in 1901 where it is desired, the fact being clearly stated on the report that the A. O. A. C. method has been employed and not that of the I. A. L. T. C." Also, at this meeting the following resolution was adopted: "(E) Used liquors must be analyzed by the A. O. A. C. method of 1901." The history of the American Associations discloses no such parallel of discontent with the accepted method, but a harmonious and successful effort to build up a method upon well-established foundations. That the members of the International Association are even at the present time far from satisfied with their official method is plainly evinced by the numerous objections that have been raised to it in recent utterances. This may be seen by consulting the files of the "Collegium"—the official organ of the I. A. L. T. C., and particular attention is directed to the paper pre-

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sented by Ferdinand Kopecky upon "The Chromed Hide Powder Question" (Collegium, 1906, Nos. 199 to 203), and the article from the pen of Prof. Procter (Collegium, 1906, No. 173), a quotation from which is here given: "While I do not wish to pose at all as of particular authority on the matter of the hide powder filter, it may be borne in mind that as its inventor I am not likely to be prejudiced against it, and that my experience of the use of hide powder for gravimetric analysis dates from its very earliest introduction, and that therefore if I conclude that this particular mode of manipulation has served its purpose and ought now to be superseded, my opinion is entitled at least to rather careful consideration."

In comparing the two methods there are two main points to be considered, the means by which the Soluble Solids content and the Non-tannin content are estimated. In all other particulars the two methods are essentially the same, and more especially in the strength of the solutions used for the analysis which calls for a dilution containing 0.35 to 0.45 grams tannin per 100 cc. in each case. On account of the agreement in the strength of the solutions analyzed the two methods are more readily comparable in those particulars wherein they disagree, since it is universally conceded that where stronger or weaker dilutions are used the tannin content will vary, largely owing to differences in the amounts of Insoluble matters. Therefore, this essay will consider the "relative value" of the two methods "as an expression of the actual value of tanning materials employed in the manufacture of leather" on the basis of equivalent dilutions, both expressing the actual value of the material analyzed, at the given strength.

SOLUBLE SOLIDS.

The two methods of filtration for Soluble Solids will first be considered. The American method calls for filtration through No. 590 S. & S., single-pleated filter paper with the aid of kaolin (free from soluble salts) previously washed in a separate portion of the tannin solution. The paper used is a fine, *quantitative* paper, which has been proved to absorb less tannin than any other tested. A stated portion, amounting to 150 cc. of the filtrate, is discarded before collecting the 100 cc. used for the determination, it being proved that the 150 cc., to a large extent but not altogether,

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satisfies the affinity of the paper for tannin. The I. A. L. T. C. method is one proposed by Dr. J. Gordon Parker and E. E. Munro Payne (Collegium, 1904, No. 120, page 249), and later adopted as official by the executive committee, as empowered by the Seventh Conference of the I. A. L. T. C., held at Turin, from the results obtained by a commission of twelve appointed at the said conference. (Collegium, 1905, No. 143).

Briefly stated, the method calls for filtration of tannin solutions through an especially devised candle filter, under reduced pressure. The Insolubles of the solution are presumed to remain on the exterior of the candle, an aliquot portion of the solution passing through, being evaporated for the soluble solids determination. The I. A. L. T. C. method was given exhaustive study by the American Association, and the results will be found in the Journal of the American Leather Chemists' Association, Vol. I., No. 1, pages 43 to 54, inclusive. The method of candle filtration is here proved to be absolutely unreliable, as the following table of averages will show:—

I. A. L. T. C. Candle Filter Method vs. A. O. A. C. and A. L. C. A. Paper Filter Method.

	Insolubles			
	Quebracho Extract Ordinary		Hemlock Extract	
	A.O.A.C. & A.L.C.A. Paper	I.A.L.T.C. Candle Filter	A.O.A.C. & A.L.C.A. Paper	I.A.L.T.C. Candle Filter
Average.....	7.56	5.75	3.90	5.11
Maximum.....	8.65	10.85	4.87	14.25
Minimum.....	6.37	.45	2.54	1.30
Greatest Difference	2.27	10.40	2.33	12.95

The filter candles used were obtained through Dr. J. Gordon Parker. The number of analysts participating in the comparative work was sixteen, and without a single exception, they condemned the candle filter method. So high an authority in the International Association as Prof. Procter has affirmed that the method leads to erroneous results. He says, (Collegium, 1906, No. 209, page 173): "When, however, we come to the question of filtration, and the determination of so-called 'insolubles,' I feel that some change in our present rule is urgently required. Whether owing to some variation in the porosity of the candles or to difference in the extracts which come before us, the expectation which I think was held by everyone, that practically clear filtrates

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would be obtained, when they accepted my resolution that 'substances passing through the candle should be considered as soluble,' has not been fulfilled. Certain extracts, especially Quebracho extracts, pass through the candle, even after filtering considerable quantities, in a very turbid condition, and the analytical results naturally vary in a very marked manner from those obtained by our previous method of filtration with paper and the use of kaolin, even after adding the correction which is requisite for tannin absorbed by the filtering material. The following table gives a few instances of this which have occurred in my own laboratory:—

QUEBRACHO EXTRACTS (INSOLUBLES)

Marked	"549"	"1142"	"C.F.H."	"1259"	"1261"	"1249"	"1248"
I.A.L.T.C. Candle method (Turbid)	2.3	4.2	1.0	4.4	4.4	3.8	2.5
605 S.S. Kaolin and Correction (Clear)	10.0	11.5	8.9	11.1	12.9	11.8	13.7

In some of these cases the inaccuracy of the analysis has been so obvious that I have been compelled to report according to the older method, pointing out in the report that it was impossible to obtain a suitable filtrate with the candle."

It is not the purpose of this essay to dwell to any extreme extent upon the differences obtained by the two methods owing to variation in methods of soluble solids estimation. It was thought best, however, not to pass over the subject without comment, inasmuch as it is to a degree as much a part and parcel of the analysis as is the determination of non-tannin substance. However, attention is called to the fact that the commission reporting in favor of the candle filter based its decision upon results obtained from it in comparison with filtration through "S. & S., No. 605 hard" paper, used with a correction factor as called for by the I. A. L. T. C. method previous to the adoption of the candle filter.

K. Schorlemmer (Collegium, 1904, 137-144), has pointed out that this "correction factor" is in itself often erroneous owing to variability in the paper itself and the character of the solution filtered, and concludes that equally true results might be obtained more simply by rejecting a larger quantity of filtrate before collecting for evaporation, and discarding the correction factor, which would amount practically to the American method.

It is therefore pointed out that although the commission de-

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cided in favor of the Candle Filter, it did so in comparison with a method which is in itself erroneous. In this connection it is interesting to note that the A. O. A. C. experimented with the "correction factor" in 1903, (U. S. Department of Agriculture, 1903, Bulletin No. 81, page 223), but condemned it on the ground that "the method employed to ascertain this correction augments to an exaggerated extent the error already existing in the determination of soluble solids."

It is argued, therefore, that the American Method for Soluble Solids Determination, although undoubtedly giving some slight error owing to tannin absorption by filter paper, nevertheless is immensely superior to the present candle filter method of the International Association, both in the concordance of the results obtained and as an expression of the actual value of the tannin material.

NON-TANNIN DETERMINATIONS.

It is upon the point of the divergency in non-tannin results obtained by the Shake and Filter Method that the essay would lay particular stress. The American Shake Method specifies that a lightly chromed, wet hide powder, containing from 70% to 75% of moisture and of an amount equivalent to from 12 grams to 13 grams of dried powder, shall be shaken in a mechanical shaker with 200 cc. of the tannin solution for ten minutes, the magma squeezed by hand through linen, the detannized solution filtered through filter paper with the aid of kaolin and 100 cc. of the clear filtrate evaporated for the determination. The result is to be corrected for the amount of water contained in the wet hide powder. The International Method calls for the upward filtration of the tannin solution through dry, unchromed hide powder packed in an inverted filter tube, the filtrate being rejected so long as it contains soluble hide, which is practically contained in the first 30 cc. The next 50 cc. filtering over are used for the non-tannin determination.

Invariably the results obtained from the filter method show lower non-tannins than when the same tanning material is analyzed by the Shake Method. Which result is nearer the actual value of the tanning materials? Why should there be this difference in non-tannins?

It is recognized both by the leather manufacturer and the chem-

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ist that hide has an affinity for matters not tannin as well as matters properly tannin. Does this phenomenon of non-tannin absorption reflect itself in the analysis, and does it reflect itself to a greater extent in the one method than in the other? Surely, if one method returns less non-tannin matters as tannin than the other, that method expresses more nearly the actual value of the tanning material, unless we assume that non-tannins have tanning properties which is an absurdity.

Therefore it devolves upon this essay to prove, if possible, whether the Shake Method or the Filter Method absorbs the greater amount of non-tannin matters.

Reference is here made to the communication from Prof. H. R. Procter and F. A. Blockey, (Journal Society Chemical Industry, April 30th, 1903, page 482), in which the absorption of non-tannin matters is discussed at considerable length. To quote: "The results, which are tabulated in this paper, show clearly that the error is a very serious one, and that it is common to all the present methods in which hide powder is used, though in most cases the shaking methods are less affected than by filtration." ———— ————"Where gallic acid is present in considerable quantities, as in sumac and myrobalans, much truer results will be obtained by acting on the resolution of the International Association permitting the use of the chromed hide powder method on condition that its use is stated in the report, and in the case of used and acid liquors it is imperative that this should be done. In this connection it is only fair to state that the contention of the American Association of Official Agricultural Chemists as to the superior accuracy of the shake method, and that of the Vienna Research Institute as to that of the use of chromed hide powder are fully confirmed by these experiments."

Freiburg hide powder was in this research used in the shake as well as the filter method. Freiburg powder is not suitable for use in the shake method as the author has determined many times in his laboratory, and as has been confirmed by other analysts. It is impossible to quote at any length the results obtained by the estimation of a tanning material before and after the addition of various non-tanning substances, but it is urgently desired that the article in question be consulted.

The author considers that in conducting the experiments Proc-

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ter and Blockey employed too large a proportion of non-tanning matters, thereby increasing the amount of non-tannins to an extent that does not obtain in the usual run of materials analyzed. With this idea he has made analyses along the lines suggested by Procter and Blockey, but with a smaller proportion of added non-tanning substances. The results are shown in the following tables. Freiburg hide powder was used for the filter method, and hide powder made in America for the shake method. The Freiburg powder was of inferior quality yielding more soluble matters than it should, which would tend to increase the non-tannins and produce results more favorable to the filter method than is actually the case.

Other experiments were conducted along the same lines with slightly larger proportion of the gallotannic acid, as shown in Table III., but in the case of the Quebracho extract and its admixtures with non-tanning substances the same solutions used in Table II. were employed. In Tables III. and IV. a better grade of hide powder was used for the filter method.

It is very evident from the tables that the absorption of added non-tanning matters in the filter method far exceeds that in the shake method, hence it follows that the natural non-tannin matters present in a tanning material are absorbed to a greater extent in the former than the latter method. Nor is the explanation a complicated one. In the shake method the entire quantity of hide powder used is added to the entire quantity of tanning solution to be detannized. The tannin present acting more rapidly upon the hide powder than the non-tannin is absorbed by it, tans the exterior of the hide particles, thus preventing access of non-tannin to the interior and untanned portions. With the filter method the effect of the procedure is quite different. Here the tanning solution is added slowly into the beaker in which the filter tube is placed, in order that it may be absorbed and rise in the tube by capillary attraction so as to prevent the forming of channels and a too rapid tannage of the powder. When the filtration is conducted it is approximately at the rate of a drop in two seconds, and this slow filtration, together with the slow addition of the solution previous to filtration, permits a very thorough tannage of the hide powder in the base of the filter tube, leaving that in the upper portion of the tube almost entirely unacted upon by

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TABLE I.

Showing Absorption of Non-Tanning Substances by Shake and Filter Methods.

Materials.	Gms. per Litre.	Total Solids.	Soluble Solids.	Insolubles.	Non-Tannins.		Tannins.		Excess of Tannin from Filter over Shake.	Gain or Loss per cent. in Tannin from Added Substance.		Percentage of Dry Non-Tannin Substance Estimated as Tannin.	
					Shake.	Filter-Tube.	Shake.	Filter-Tube.		Shake.	Filter-Tube.	Shake.	Filter-Tube.
Gallotannic Acid.....	4.5	88.91	88.69	0.22	8.57	7.15	80.12	81.54	1.42	.....	.....	.....	.....
Gallotannic Acid.....	4.5												
Glucose	1.0	109.11	108.94	0.17	28.92	27.66	80.02	81.28	1.26	—0.10	—0.26	—0.50	—1.28
Gallotannic Acid.....	4.5												
Gallic Acid	1.0	108.91	108.84	0.07	18.75	11.84	90.09	97.00	6.91	+9.97	+15.46	+49.48	+76.73
Gallotannic Acid.....	4.5												
Dextrine	1.0	108.71	108.42	0.29	29.18	24.69	79.24	83.73	4.49	—0.88	+2.19	—4.46	+11.10
Gallotannic Acid.....	4.5												
Glucose ⅓ gm. ...													
Gallic Acid ⅓ gm.	1.0	109.04	108.34	0.70	25.54	21.11	82.80	87.23	4.43	+2.68	+5.69	+13.64	+28.96
Dextrine ⅓ gm. ..													

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TABLE II.

Showing Absorption of Non-Tanning Substances by Shake and Filter Methods

Material	Gms. per Liter	Total Solids	Soluble Solids	Insolubles	Non-Tannins		Tannins		Excess of Tannin from Filter over Shake	Gain or Loss per cent. in Tannin from added Substance		Percentage of dry Non-Tannin Substance estimated as Tannin	
					Shake	Filter Tube	Shake	Filter Tube		Shake	Filter Tube	Shake	Filter Tube
Quebracho Extract, (Solid, Ordinary)	6.0	84.71	77.68	7.03	12.07	11.74	65.61	65.94	0.33	....	....	....	....
Quebracho Extract, (Solid, Ordinary) 6.0 Glucose 1.0		100.35	93.95	6.40	28.72	26.64	65.23	67.31	2.08	—0.38	+1.37	— 2.36	+ 8.42
Quebracho Extract, (Solid, Ordinary) 6.0 Gallic Acid 1.0		99.85	91.32	8.53	19.18	18.12	72.14	73.20	1.06	+6.53	+7.26	+47.87	+53.23
Quebracho Extract. (Solid, Ordinary) 6.0 Dextrine 1.0		101.22	94.30	6.92	28.63	28.14	65.67	66.16	0.49	+0.06	+0.22	+ 0.36	+ 1.33
Quebracho Extract, (Solid, Ordinary) 6.0 Glucose, 1/3 gm. Gallic Acid, 1/3 gm. Dextrine, 1/3 gm.	1.0	100.25	91.70	8.55	25.11	23.90	66.59	67.80	1.21	+0.98	+1.86	+ 7.00	+13.27

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TABLE III.

Showing Absorption of Non-Tanning Substances By Shake and Filter Methods.

Material.	Gms. per Litre.	Total Solids.	Soluble Solids.	Insolubles.	Non-Tannins. Shake.	Non-Tannins. Filter-Tube.	Tannins. Shake.	Tannins. Filter-Tube.	Excess of Tannin from Filter over Shake.	Gain or Loss per cent. in Tannin from Added Substance. Shake.	Gain or Loss per cent. in Tannin from Added Substance. Filter-Tube.	Percentage of Dry Non-Tannin Substance Estimated as Tannin. Shake.	Percentage of Dry Non-Tannin Substance Estimated as Tannin. Filter-Tube.
Gallotannic Acid.....	5.0	90.14	89.89	0.25	9.72	3.61	80.17	86.28	6.11	.....	.....	.....	.....
Gallotannic Acid..... Glucose	5.0 1.0	110.12	109.57	0.55	28.55	23.67	81.02	85.90	4.88	+0.85	—0.38	+4.32	—1.94
Gallotannic Acid..... Gallic Acid.....	5.0 1.0	109.96	108.90	1.06	19.74	3.87	89.16	105.03	15.87	+8.99	+18.75	+47.29	+98.63
Gallotannic Acid..... Dextrine	5.0 1.0	109.72	108.94	0.78	28.33	19.17	80.61	89.77	9.16	+0.44	+3.49	+2.31	+18.32
Gallotannic Acid..... Glucose ⅓ gm. ... Gallic Acid ⅓ gm. Dextrine ⅓ gm. ..	5.0 1.0	110.31	109.53	0.78	25.17	14.98	84.36	94.55	10.19	+3.75	+8.27	+19.10	+42.11

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TABLE IV.

Showing Absorption of Non-Tanning Substances by Shake and Filter Methods

Material	Gms. per Liter	Total Solids	Soluble Solids	Non-Tannins		Tannins		Excess of Tannin from Filter over Shake	Gain or Loss per cent. in Tannin from added Substance		Percentage of dry Non-Tannin Substance estimated as Tannin	
				Insolubles	Shake	Filter-Tube	Shake	Filter-Tube		Shake	Filter Tube	Shake	Filter-Tube
Quebracho Extract, (Solid, Ordinary)...	6.0	84.71	77.68	7.03	12.07	8.32	65.61	69.36	3.75	....	....	....	....
Quebracho Extract, (Solid, Ordinary)... 6.0 Glucose	1.0	100.35	93.95	6.40	28.72	21.20	65.23	72.75	7.52	—0.38	+ 3.39	— 2.36	+20.84
Quebracho Extract, (Solid, Ordinary)... 6.0 Gallic Acid	1.0	99.85	91.32	8.53	19.18	11.74	72.14	79.58	7.44	+6.53	+10.22	+47.87	+74.93
Quebracho Extract, (Solid, Ordinary)... 6.0 Dextrine	1.0	101.22	94.30	6.92	28.63	23.20	65.67	71.11	5.44	+0.06	+ 1.75	+ 0.36	+10.53
Quebracho Extract, (Soiid, Ordinary)... 6.0 Glucose, 1/3 gm. Gallic Acid, 1/3 gm. Dextrine, 1/3 gm.	1.0	100.25	91.70	8.55	25.11	18.80	66.59	72.90	6.31	+0.98	+ 3.54	+ 7.00	+25.25

NOTE.—It will be observed that an interaction has taken place in several instances between the added non-tanning substance and the tanning material. This is most plainly shown by differences in the "Soluble Solids" and "Insolubles" items. No account of this has been taken, however, as the comparison of the two methods is not affected.

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the tannin, and therefore in a condition to absorb a large amount of non-tannin matters. If the filtration is hastened tannin matter is liable to filter over, and the rate of filtration determines the non-tannin content.

Summarized, the points that lead to divergent results in the filter tube method are as follows:—

1. *The Shape of the Filter Tube:* The narrower and taller the tube the larger the quantity of hide powder through which the solution must pass, and the greater the non-tannin absorption, and *vice versa.* As this depends upon apparatus it is not paralleled in the shake method.

2. *Speed of Filtration:* The more rapid the filtration the greater the proportion of the hide powder tanned and the less the proportion of unacted upon powder left to absorb non-tannins, and *vice versa.* With the shake method the time of shaking has almost no effect upon the result.

This is shown by the A. L. C. A. Report of 1905, (Journal American Leather Chemists Association, Vol. I., No. 1, page 62), where tannery liquors rich in non-tannins gave results shown in the following table:—

NON-TANNINS.

RESULTS OBTAINED ON LIQUORS BY VARIATION IN TIME OF SHAKING.

Barkometer Degree	18.4		17.1	
Non-Tannins, 10 minutes shake...	4.127	4.210	3.814	3.867
Non-Tannins, 30 minutes shake...	4.126	4.202	3.821	3.871
Amount Dry Hide represented....	4.43 gms.	2.65 gms.	4.43 gms.	2.65 gms.

This is also proved by the following figures obtained from one of the largest laboratores connected with the [illegible]ner industry in the United States.

TANNINS. CHESTNUT EXTRACT.

RESULTS OBTAINED BY VARIATION IN TIME OF SHAKING FOR NON-TANNIN ESTIMATION.

	Shaken for 5 Min.	10 Min.	15 Min.	20 Min.
25 gms. Wet Hide.....	26.19	25.99	26.02	26.18
30 gms. Wet Hide.....	26.08	26.03	26.10	26.08
35 gms. Wet Hide.....	25.99	25.99	25.72	25.54
40 gms. Wet Hide.....	25.87	25.88	25.64	25.47
45 gms. Wet Hide.....	25.55	25.96	25.50	25.72
50 gms. Wet Hide.....	25.54	25.28	25.57	25.77

As for the effect of the speed of shaking upon the non-tannin figure, the author would state that the mechanical shaker formerly used in his laboratory, was the ordinary milk shaker run by

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power, and holding but two shaker glasses and running at a speed of over 200 revolutions per minute. Finding its capacity insufficient he had built a shaker holding twelve-quart bottles, six on either side of a central axis, and running at the rate of from 60 to 65 revolutions per minute on the tumbling system. Exactly the same non-tannin results are obtained from this shaker as from the old style milk shaker.

3. *Manner in which Tube is Packed:* If the powder is too loosely packed in the tube channels will form and tannin appear in the filtrate, and if too tightly packed the filtration is retarded, one leading to high and the other to low non-tannin figures. In this respect no comparison can be made with the shake method.

4. *Character of the Hide Powder:* Hide powder, if a trifle too acid is unfit for use in the filter tube, as it swells and retards filtration. Unchromed powder will gradually give up soluble matters to the filtrate, the amount given up depending upon the character of the powder. In the shake method an acid powder will give lower non-tannins than a less acid one, but since the hide powder is used wet the degree of acidity may be controlled by neutralization during chroming or washing. Also chromed and washed powder will give up less solubles to the non-tannins.

5. *Character of Solution Analyzed:* A used (acid) liquor cannot be properly analyzed by the filter method, which is recognized by the fact of the adoption of the shake method for such liquors by the I. A. L. T. C. The absorption of non-tannin matters is greater in the case of used liquors, owing not only to the larger proportion of non-tannins present, but to the constituents of the non-tannins themselves. The acid present serves to swell the hide powder and produce a greater absorption of non-tannins than with fresh materials. This has been conceded in the American method by the adoption of a Provisional method for liquor analysis at the last meeting of the A. L. C. A. and A. O. A. C. (Journal American Leather Chemists' Association, Vol. I., No. 1, page 87) by which the amount of chromed hide powder is reduced on account of the greater absorption of non-tannins before mentioned. It is assuredly a point against the International method that it requires one method of analysis for fresh tanning materials and another for used liquors, and in the same degree it is a point in favor of the American method. It might be well to

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call attention again to the fact that Procter and Blockey recommended the shake method for materials containing considerable amounts of gallic acid. The shake method is a general method, whereas the filter method is not.

With the shake method a more acid hide powder gives lower non-tannins than a less acid one, as above stated. This is entirely a physical phenomenon. A greater amount of water in the wet hide will produce a similar result. The reason is simply that the more acid the powder the greater its swelling properties, or the more water contained the greater the bulk occupied. In each instance a larger surface of powder is exposed to the action of the tannin solution and a tannage of less depth is produced, permitting a greater penetration of non-tannin matters, and resulting in lower non-tannins. With less water or a less acid hide powder, the opposite effect is produced, and if the hide powder is neutral the absorption of non-tannins may not only be nil, but the entire quantity of tannin itself may not be fixed. The majority of dry chromed hide powders do not work well in the shake method, for the reason that they do not swell sufficiently to present the surface required to detannize. If such powders were made more acid a sufficient swelling would in all probability be produced. With acid liquors an acid hide powder is of distinct benefit to the accuracy of the results, since the acidity to a degree prevents the absorption of more acid. That the shake method does give excellent results on acid liquors is proved by the report of the Acid Committee of the A. L. C. A., 1905, (Journal American Leather Chemists' Association, Vol. I., No. 1, page 69), showing that the addition of varying quantities of acetic, lactic and sulphuric acids to a fresh hemlock bark liquor affected the tannin contents but slightly.

Again, returning to the filter method, Tables I. to IV., have proven it to be wrong in principle, since by the very nature of its manipulation detannized (or non-tannin) solutions are presented to fresh unacted upon hide powder. And if the principle is wrong the error should appear with dry chromed hide powder as well as with a dry unchromed powder, which latter has but recently been adopted by the German Section of the International Association. The author has conducted certain further experiments that prove beyond question of doubt the erroneous princi-

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ple of the filter method, both with chromed and unchromed hide powder. It had occurred to him that as the hide powder in the filter tube became saturated with the tannin and non-tannin matters there should be a gradual increase in the weight of the non-tannin filtrate. In order to prove this a number of tests were run and successive 50 cc. fractions of the filtrate coming over were evaporated for non-tannin determinations after the soluble hide had disappeared, which was practically always after the filtration of the first 30 cc. Frequent tests were made to ascertain whether tannin was appearing in the filtrate and the occurrence of tannin is noted in Table V.

This table proves very conclusively that the filter method not only does not give the correct non-tanning figure, but does not compare with the shake method in this respcet. A careful study of the results will show that as the non-tannins increase with the evaporation of the successive 50 cc. portions, they, at the same time, approach to the non-tannin figure obtained by the shake method, and that in those instances where the filtration was conducted until the appearance of tannin in the filtrate, the fraction immediately preceding its appearance approximates very closely in non-tannin percentage to that obtained from the shake method. The non-tannins from the filter method with chromed hide powder are lower than with unchromed powder, which is parallel to the results obtained by the A. O. A. C., at the time of the adoption of wet chromed hide. (Proceedings of the 19th Annual Convention A. O. A. C., Bulletin No. 67, pages 130 to 142). The difference is undoubtedly due to soluble matters from the unchromed hide powder which appear in the non-tannin filtrate. Therefore, it can be safely assumed that despite the low non-tannins obtained by the filter method (with unchromed powder) they would be even lower if it were not for the soluble hide matters contained.

That chromed hide powder in the filter tube will give lower non-tannins than unchromed, is proved also by results shown in Collegium, Nos. 207 and 208. These are compiled in the following table of averages—(Table VI).

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TABLE V.

SHOWING RESULTS IN NON-TANNIN PERCENTAGE FROM EVAPORATION OF SUCCESSIVELY FILTERED FRACTIONS FROM FILTER-TUBE.

Material.	Shake, Official.	Hide-Powder used in Filter	Filter-Tube Method, showing results from successive 50 cc. Evap. 1st	2nd	3rd	4th	5th	6th	Shake, Modified.	Filter, Theoretical.	Shake, Theoretical.
Quebracho Extract (Solid, Ordinary)	12.07	Chromed	9.67	9.93	10.26	10.76	11.78	T	....	....	....
Hemlock Extract	....	Chromed	13.91	15.69	18.09	T	....	....	....	....	....
Quebracho Extract (Liquid, Clarified) (*a*)	10.85	Chromed	9 20	10.20	10.64	11.00	T	....	....	....	....
	....	Unchromed	9.72	11.52	11.66	11.94	....	....	....	....	....
Quebracho Extract (Liquid, Clarified) (*b*)	11.56	Chromed	9.70	10.60	11.50	11.90	....	....	....	....	....
	....	Unchromed	10.80	12.30	12.50	12.82	....	....	....	....	....
Quebracho Extract (Solid, Clarified) Dry	19.13	Chromed	14.60	14.80	15.20	16.68	T	...	19.05	....	....
	....	Unchromed	15.20	15.20	15.32	16.92	T20.00	....	....	....	....
Mangrove Extract (Liquid)	14.29	Chromed	11.38	T	....	....	....	....	10.42	....	....
	....	Unchromed	12.54	T	....	....	....	....	....	....	....
Oak Bark Extract	21.49	Chromed	17.29	19.66	20.41	21.17	T	....	19.66	....	....
	....	Unchromed	16.71	19.57	20.60	21.69	T	....	....	....	....
Chestnut Oak Extract (*a*)	....	Unchromed	14.93	16.67	17.20	17.92	T	....	....	....	....
Chestnut Oak Extract (*b*)	....	Unchromed	14.13	15.47	16.43	T	....	....	....	....	....
Chestnut Extract	14.86	Chromed	10.95	12.26	12.70	....	....	....	....	....	....
	....	Unchromed	10.87	12.43	13.57	13.91	14.13	T	....	....	....
Gallotannic Acid	8.57	Freiburg	7.15	7.91	8.09	....	....	....	....	....	....
Gallotannic Acid and Glucose	28.92	Freiburg	27.66	28.00	29.11	....	....	....	....	27.40	28.82
Gallotannic Acid and Gallic Acid	18.75	Freiburg	11.84	17.27	25.42	....	....	....	...	27.30	28.72
Gallotannic Acid and Dextrine	29.18	Freiburg	24.69	24.11	24.77	....	....	....	....	26.88	28.30
Gallotannic Acid, Glucose, Gallic Acid and Dextrine	25.54	Freiburg	21.11	20.80	22.58	....	....	....	....	26.80	28.22
Gallotannic Acid and Gallic Acid	19.74	Ordinary	3.87	4.69	13.23(*1*) 16.54(*2*)	....	....	....	....	22.62	28.73

T indicates appearance of Tannin in filtrate. (*1*) and (*2*) indicate duplicates, the former about one-third more rapid than latter in speed of filtration, but both within limits of method. Even within the specified limits there is liability to great error.

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TABLE VI.

SHOWING LOWER NON-TANNINS IN FILTER-TUBE FROM CHROMED POWDER THAN FROM UNCHROMED

Material	Ordinary Official	Lightly Chromed	Heavily Chromed	Heavily Chromed Slightly Acid
Quebracho Extract.....	5.4	4.2	4.1	4.0
Oakwood Extract.....	14.2	11.5	11.5	11.1
Pine Extract.....	13.8	12.7	13.2	12.1
Sumac Extract.....	20.3	16.7	16.3	14.6
Mangrove Extract	7.9	7.1	8.5	6.7
Mimosa—Renner Ideal..	6.2	5.2	5.2	5.1
Mimosa—Renner D.....	10.9	9.3	8.9	8.8
Mimosa Extract, Liquid.	10.0	9.3	9.3	9.0

It follows also that the 50 cc. portion evaporated for non-tannins in the filter method is in itself composed of fractions, dissimilar in the results they will give. This is clearly demonstrated in the following table where the two 25 cc. portions composing the 50 cc. used in the official determination are employed for non-tannin estimation.

TABLE

NON-TANNINS, FILTER METHOD, SHOWING FRACTIONAL COMPOSITION OF THE 50 CC. EVAPORATED FOR THE DETERMINATION

Material	1st 25 cc.	2nd 25 cc.	50 cc. Official
Chestnut Extract	10.67	11.07	10.87
Chestnut Oak Extract (1).....	14.93	16.67	15.80
Chestnut Oak Extract (2).....	14.13	15.47	14.80
A Gallotannic Acid	4.89	5.95	5.42
A Solid Quebracho Extract.....	12.36	13.09	12.72
Hemlock Extract	13.91	15.69	14.80

If the 50 cc. evaporated in the filter method is assumed to give the correct figure for non-tannins, then certainly any and every portion of the 50 cc. should agree. Such is not the case.

Referring to Table V., you will observe a column marked, "Shake, Modified." In this case the wet hide powder was added as follows:—

1st portion, 10 gms., shaken for 10 min., in shaker.
2nd " 10 " " " 10 " " "
3rd " 10 " " " 10 " " "
Last " 23 " " " 10 " and removed and shaken infrequently by hand for 50 min. longer.

The effect of this procedure should approximate that of the filter method, and the results prove this to be true. The first portions of hide powder remove most of the tannin leaving non-tannins to be absorbed by the fresh hide added.

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RESUMÉ.

In conclusion, the author would assert that as proved in his work and by his results submitted, the shake method, "as an expression of the actual value of tanning materials," is immensely superior to the filter method for the following reasons:—

1. In the soluble solids determination the filter-candle method of the International Association is liable to grave error and uncertainty, while the American method, although liable to a slight error owing to tannin absorption by filter paper, permits of consistent results.

2. (a) In the non-tannin determination the filter tube method by the very principle of its method allows the absorption of non-tanning substances to a degree far in excess of that from the shake method.

(b) The fractional filtrations that go to make the 50 cc. evaporated for the non-tannins in the filter method are not consistent, the said 50 cc. being but an average of the unlike fractions.

(c) The filter method is unfit for the analysis of used (acid) liquors, as admitted by the adoption of the shake method for such analyses by the I. A. L. T. C.

(d) The many minor details of the filter method, not occurring with the shake method, which, if not carefully observed, will make comparable results even more impossible to obtain.

3. The general satisfaction of the members of the American Association with the shake method, and the widespread dissatisfaction of the members of the International Association with the filter method.

One objection that has been raised to the shake method is that the occasional analyst will fine it inconvenient as compared with the filter method. Assuming this to be true (which the author does not grant) it is respectfully suggested that a method that is suitable for the analyst who has occasion to use it frequently should not be abandoned for one that is suitable to the "occasional analyst."

In conclusion, the author would lay particular stress upon the erroneous principle of the filter tube method, whether used with chromed or unchromed hide powder. If a method is wrong in principle, how can it be expected to give results that are correct?

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ADDENDUM.

As for possible improvements in the shake method the author believes these are two-fold: First, the so-called "Time-Contact" method for soluble solids filtration, suggested at the last meeting of the A. L. C. A. (Journal American Leather Chemists' Association, Vol. I., No. 1, page 41,), and secondly, some specification as to the degree of acidity of hide powder.

Respecting the first of these suggestions, the official method has already been sketched in this essay (see page 2). The *modus operandi* of the time-contact method is as follows: Stir up a portion of solution with 1 gm. kaolin, pour on No. 590 S. & S., 15 cm. pleated filter paper, fill funnel and keep full by returning filtrate for one hour. Decant solution from funnel, drain, fill with fresh solution, discard first 10 cc. of filtrate, or until clear, and collect the 100 cc. for determination.

A table is submitted below in order to show the superiority of the suggested method over the official. The two extracts selected are probably as difficult with which to obtain consistent results as can be found.

TABLE SHOWING COMPARISON OF OFFICIAL AND TIME-CONTACT METHODS FOR SOLUBLE SOLIDS DETERMINATION.

Material.	Method.	No.	Total Solids.	Soluble Solids.	Insolubles.	Appearance of Filtrate.
Quebracho Extract	Official	1	88.49	80.47	8.02	Turbid
		2	88.68	80.08	8.60	Clera
Solid, Ordinary	Time-Contact	1	88.49	79.93	8.56	Clear
		2	88.68	80.06	8.62	Clear
Hemlock Extract	Official	1	49.81	46.61	3.20	Turbid
		2	49.71	46.11	3.60	Turbid
Liquid	Time-Contact	1	49.81	45.29	4.52	Clear
		2	49.71	45.19	4.52	Clear

Analyses marked (1) were performed on the same day with identical solutions, and those marked (2) on the same day and with identical solutions, but a lapse of a week intervened between analyses (1) and (2). The insolubles figures best show the superior concordance of the time-contact method.

Regarding the specification as to the degree of hide powder acidity, once the proper degree of acidity is determined one of

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two courses should be pursued. The manufacturer of the hide powder should either produce a product of the proper acidity, or a neutral powder which the analyst could himself acidify as directed. As the latter course would entail additional labor, the former would seem to be preferable. If the manufacturer can make up a year's supply of the hide powder with the specified acid content, discrepancies in non-tannin results must be laid at the door solely of personal error.

With the improvements suggested the American shake method will occupy a position from which it can be dislodged only with the greatest difficulty.

NOTE.—The above essay was entered in competition for the Klipstein Prize, offered at the Turin Conference in 1904, but was not admitted by those appointed to adjudge the award, as failing to comply with the condition prescribed as to the time of entry.

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ANNUAL MEETING

The annual meeting of the Association will be held in New York City, November 22, 23 and 24.

ELECTIONS

ACTIVE.

Dr. J. Gordon Parker, % Herold's Institute, Bermondsey, London S. E., England.

ASSOCIATE.

Jos. M. Romadka % J. B. Ford Co., Wyandotte, Mich.

Dr. Parker has been appointed Corresponding Secretary of the Association for Europe.

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THE COMING CONVENTION.

A pleasant and profitable time can be promised the members attending our next annual meeting, and any sacrifices necessary for the attendance of those interested will be warranted by the benefits to be derived. The New York meeting will be the first convention to be held independently, and its success may be taken as a criterion by which the strength and permanency of the Association can be judged. If for no other reason than this, the active and associate membership should be well represented on the register and on the program. It is not the intention of the Association to confine the topics of discussion at our conventions, to tannin and kindred analyses, but to include for treatment, subjects and problems of practical tanning which involve chemistry more broadly applied. Our associate members must aid us in the successful presentation of these subjects, and by introducing for discussion, problems which have come up in their field of work, may profitably increase this feature of our meetings.

The social side of the meeting has not been forgotten, and at the banquet we may expect entertainment and instruction from the mouths of orators, selected from the captains of the American leather industry.

VIII. MEETING OF THE INTERNATIONAL ASSOCIATION OF LEATHER TRADES CHEMISTS HELD AT FRANKFORT a/M. SEPTEMBER 17th TO 20th, 1906.

[Collegium, 1906, pp. 329-38.]

At the meeting of the International Association of Leather Trades Chemists held at Frankfort a/M., September 17-20, Mr. Seymour-Jones, of Wrexham, announced the decision of the judges as to the Klipstein prize. The essay, "Chromhautpulver," is awarded the first prize, the authors of the same being Messrs. Dr. Paessler and W. Appelius, of Freiberg. The work of Mr. Kopecky, as well as that of Mr. Reed (*Lux et Veritas*), deserve honorable mention, although they could not be considered as they did not completely comply with all the prescribed conditions. The second prize is not awarded, although the work of Prof. Wisli-

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cenus deserves honorable mention, though also not fully complying with the prescribed conditions. The Seymour-Jones prize is this time awarded to Mr. Kopecky, of Leeds, for his essay on "The Hide Powder Question."

The meeting then took up the question of tannin analysis. Prof. Procter thoroughly describes the present conditions, and especially speaks about the disadvantages of the filter tube method. He makes the motion, in case there are no agreements, to abolish the filter tube method from January 1st, 1907, on, and to replace the same by one which is up-to-date. The motion is seconded by Schorlemmer, of Worms.

Hereupon a lively discussion ensues in which Messrs. Dr. Parker, Eitner, Jean, Dr. Lepetit, Prof. Wislicenus, Dr. Turnbull, Dr. Becker, Dr. Paessler and Nihoul join and in which especially the three last-named speakers express serious doubts as to the advisability of so hasty a change. Reed, of America, gives a demonstration of the American shake method. Mueller, of Benrath, makes the motion "That the I. A. L. T. C. should resolve that reports on tannin determinations should only be allowed to be issued when the given values are the average of two well agreeing separate analyses." Prof. Procter expresses the wish that a closer relationship will exist between The International Association of Leather Trades Chemists and The American Leather Chemists Association and that the experiences and literary productions of the latter be exchanged with them. The convention gladly agrees to this proposition and instructs the President to take steps in this direction.

Dr. Baum gives an interesting description of experiments made in determining tannin electrolytically.

Prof. Procter withdraws his motion to have the filter tube method abolished after January 1st, 1907.

In the name of the Executive Committee, Dr. Parker then makes the following motion: "The Convention shall decide to appoint a committee which shall test the different methods for the analysis of tannin by comparative tests and to report its findings to the Executive Committee by April 1st, 1907, at the latest. The Executive Committee to be instructed, after consulting again with the Commission, if necessary, to publish the method which the

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Commission finds is the best and which shall be in future the official method for the members of the I. A. L. T. C.

This Commission shall have the right to enlist other assistance and shall consist of the following gentlemen: For Scandinavia, Boegh; for Italy, Dr. Baldracco; for France, Meunier; for Belgium, Nihoul; for Germany, Dr. Klenck; for America, Reed; for Austria, Eitner; for England, Dr. Parker; and as President, Prof. Procter.

The Convention distinctly declare that they do not insist upon the retention of the filter tube."

Being put to vote both motions are unanimously carried.

Mr. Reed then gives a short description of the manipulation of the American method for determining tannin. He speaks of a new filter method, the so-called "Time-Contact Method," with which extensive experiments are now being carried on in America. He speaks of a combined evaporator and dryer by which they have obtained good results, and also calls attention to a new method for the determination of acid in tan liquors which was published by Hoppenstedt in No. 4, of *The Journal of the American Leather Chemists Association.* Dr. Becker thanks Mr. Reed for his descriptions which help to further stimulate the appointed commission.

Prof. Procter suggests to determine the insolubles by a different method than heretofore, namely at a higher temperature, as he was able to tan skin well with the reds filtered off with the present method. One should discriminate between "insoluble" and "slightly soluble" tannins.

Dr. Paessler gives an account of a new method for determining the kind of tannin in extracts, after having done a large amount of color tests. A lively discussion follows this interesting report, and at the end of which Dr. Becker states that our knowledge at the present time has not reached the height so that a chemist can determine accurately the tannin present in mixtures. Prof. Procter agrees with Dr. Becker and calls attention to the fact that there are extracts now on the market, which are made from pine wood by treating the same with sulfites, and that the same really do not contain any tannin at all, but in connection with tannin have leather forming properties.

Dr. Parker makes the motion: "Although the Convention ap-

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proves the work of Dr. Paessler, they are still of the opinion that one cannot determine with accuracy today the different constituents in mixtures of tannins." The motion was unanimously carried.

Mr. Seymour-Jones suggests to the extract manufacturers, that they should give their products a more exact specification, namely, to declare if the extract is decolorized and by what, if it is treated with bisulfite, if it is a pure extract or a mixture, etc.

Mr. E. C. Klipstein announces that he will maintain the unsolved prize competition and that the conditions regarding the same will later be published in the *Collegium* by a commission consisting of Messrs. Procter, Lepetit and Schorlemmer.

The President thanks Mr. Klipstein and also Mr. Seymour-Jones for their interest which they have shown in the Association by the offering of prizes.

Dr. Parker then speaks on the influence of chlorides, on the tanning and plumping of hides, in tan liquors and emphasizes that the chlorides do not have any detrimental action when sufficient acid is present. His experience is based on experiments on a large scale as well as in the laboratory with hide powder.

Shortly hereafter the meeting comes to a close. Dr. Becker thanks all members for making the Convention a success. Dr. Parker thanks the President for his excellent arrangements and for so ably conducting the Conference. All present join in a rising vote of thanks. Mr. Eachus, of New York, also adds a few words of thanks in the name of his American colleagues.

The Convention is then adjourned to meet again in two years at Brussels.

A METHOD FOR THE EXAMINATION OF MARINE OILS.[1]

PART I.

By H. R. Procter and H. G. Bennett.

The authors seek to gain further information regarding the nature of various marine oils by means of the "hexa-bromide test."[2]

[1] Condensed from the *J. S. C. I.*

[2] Hehner & Mitchell, *Analyst*, 1898, 313.

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After considerable investigation the following modified method was found to give the best results:

Cod Oil.—0.4 gram (approximately) is weighed into a small tared flask and 10 cc. of carbon tetra-chloride added, and 12 drops of bromine, the mixture being kept cool for three hours in running water. Remove the excess of bromine by adding 10 cc. of carbon tetra-chloride containing 0.075 gram of phenol, and add 20 cc. of absolute alcohol, gradually, with constant stirring. Filter, and allow to drain, wash with 50 cc. absolute alcohol, dry in air, place in a weighing bottle, which has been first tared, along with the paper, and heat in steam oven for a quarter of an hour; cool in desiccator and weigh. The bromine should be dropped from a tap funnel at a rate convenient for counting, and with duplicate experiments, should drop at the same rate.

For any given oil it is necessary to keep the weight taken, very nearly the same, for, if too little be taken, more bromine will be left over than will be de-brominated by the small quantity of phenol added, and if too much be taken, there may be no bromine left in excess. With these precautions and some care, results varying within 1 per cent. can be obtained. This percentage yield of bromides is called the "bromide value."[1]

Bromide value.	Bromine Percentage.
(1) 60.9	65.7
(2) 60.0	63.3
(3) 60.4	63.5
(4) 60.8	63.7

As the quantities of reagents are made definite by the method in applying this to other oils, it is necessary to take quantities which will give the same amount of unsaturation. These quantities can be obtained by making them inversely proportional to the iodine value. In filtering, it is desirable, for the sake of accuracy, to keep the filter full.

The method applied to the fatty acids from cod oil gave a low bromide value, 10.4, showing that an oil of high acid value will not have its bromide value seriously affected.

The results of the method applied to various oils are given below.

From the iodine value given for an oil, the "bromine value" can

[1] Brown Cod oil (iodine value, 150.7.)

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be calculated, and from this and the weight of the oil taken, the total amount of bromine absorbed by this amount of oil is obtained, and as the weight of bromine in the precipitate is determined, it is possible to find how much of the total bromine was absorbed by the glycerides, which give the precipitate, and how much by those which do not. This distribution is best expressed in percentage of the total bromine absorbed.

Example:—0.4138 gram of cod oil gave 0.2502 gram of precipitate containing 0.1591 gram of bromine. From the bromine value it is seen that 0.4138 gram of the oil absorbs 0.3927 gram of bromine.

The percentage of total bromine absorbed which is taken up by the glycerides giving precipitates:

$$= \frac{0.1591 \times 100}{0.3927} = 40.5.$$

Assuming that the formation of these bromides is due merely to the addition of bromine to the unsaturated linkages in the oils, the weight of the glycerides which gave the precipitate should be obtained by subtracting the weight of bromine found in the precipitate from the weight of precipitate taken.

The bromine value of these glycerides may therefore be calculated, and by use of the factor, 127/80, it is possible to obtain the iodine value of the more highly unsaturated portions of the oil. If these portions are the most readily oxidizable, this figure may be of more value than the iodine value for indicating the quality and state of oxidation.

The authors hope that after the range of variation of the bromide value has been observed in a series of oils, that it will be of value in detecting adulterations.

The bromine in the precipitates was determined by hydrolyzing in the water bath for 3 hours, by the action of alcoholic potash, washing into a calibrated flask, acidifying with formic acid, neutralizing with magnesium oxide and making up to the mark with distilled water. The insoluble portions filtered off, and the potassium bromide estimated in aliquot parts of the filtrate with N/10 silver nitrate and potassium chromate indicator. From the percentage of bromine obtained in precipitates from brown cod oil, the authors assume that the precipitates must be either the bro-

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mide of a mixed glyceride or a hexa-bromide with lower bromides mixed with it, or both.

By taking a larger amount of brown cod oil and carbon tetrachloride and brominating and collecting fractions of the precipitate formed by the addition of successive quantities of alcohol, it was found by estimation of the bromine contained in the several fractions that, roughly speaking, the "hexa-bromide" is first precipitated, followed successively by the tetra-bromide, the dibromide, and saturated oil.

A further fractionation of the first precipitate resulted in obtaining a compound corresponding to the "octa-bromide."

OIL.	Iodine value.	Bromine value by calculation.	Bromide value.	Percentage of bromine in precipitate.	Percentage of total bromine absorbed, taken up by the glycerides giving the precipitate.	Mean iodine value of glycerides giving precipitate.
Brown Cod	150.7	94.9	60.4 60.8 60.0	63.5 63.7 63.3	40.5 40.8 40.1	277*
Newfoundland Cod	141	88.8	49.5 48.6	52.4 55.3	29.1 30.3	185
Möller's Cod	163.2	102.8	41.6 42.7	60.9 62.6	24.2 26.1	267
Möller's Cod ... (blown 24 hours)	147.0	92.60	30.2 28.7	58.8 62.0	19.2 19.1	243
Linseed	158.7	100.0	24.8 25.0 24.5	61.5	15.5	253
Menhaden..... (marked "4")	154.2	97.1	52.7 54.7 52.6	62.4 61.7 52.2	33.9 34.5 28.3	258
Menhaden..... (marked "5")	157.3	99.4	60.5 59.4	58.8 62.4	36.9 37.3	245
Pale Seal	102.5	64.5	13.2 15.0	51.3	11.2	147
Dark Seal	90.6	57.0	13.4 14.5	60.0	13.7	238
Pale Whale.....	120.7	76.0	27.5 27.2	58.4 62.1	21.1 22.2	242
Dark Whale	114.2	71.9	37.0 37.8	59.6 60.3	28.0 31.4	239
Shark's Liver.....	109.7	69.1	15.4 17.6	55.4 56.3	14.1 14.3	205
Sperm.....	81.8	51.5	6.5 6.2	45.7	5.6	134
Coal Fish.....	130.7	82.3	29.9 29.6	58.9 56.2	21.4 20.5	216
Fresh Herring	136.4	85.9	44.8 44.7	47.7 55.5	24.9 27.2	180

* The glycerides giving the "octa-bromides" recorded in the fractionation experiments have an iodine value of 380 approximately.

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QUALITATIVE TANNIN ANALYSIS.

M. Nierenstein, in recent European publications, shows some interesting reactions with diazobenzene chloride. One gram of quebracho tannin was dissolved in 500 cc. of water, and the solution in one case mixed with 100 grams of sodium acetate, and in another case without such addition, was cooled with ice, and a one-half per cent. solution of diazobenzene chloride added drop by drop until a turbidity was no longer produced. After standing for 24 hours, the deep red precipitate was filtered off, and repeatedly boiled with acetone, the color changing to light red during this treatement. The azo compound thus obtained was insoluble in alcohol and other solvents, and did not melt below 360° C. It contains from 52.2 to 59.9 per cent. of carbon, 2.97 to 4 per cent of hydrogen, and 13.17 to 14.55 per cent. of nitrogen. By extracting the reaction product with absolute alcohol, and evaporating the solution, a red powder was obtained, which had all the properties of a catechol tannin, but was optically inactive, whereas, quebracho tannin has, according to Trimble, a rotating power of from + 11° to + 26°. This indicates that the optical activity of a tannin substance may be due to the accompanying sugar, rather than to the tannin.

Not only quebracho tannin, but all pyrocatechol tanning matters yield a precipitate with diazobenzene chloride, whereas no pyrogallol tannin gives the reaction. The reaction is carried out by adding to the cold solution of the tanning material, drop by drop, a one-half per cent. solution of diazobenzene chloride. The precipitate appears at once. The solution of the reagent may be kept unchanged for months in a brown glass bottle.

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DETERMINATION OF SULPHURIC ACID IN LEATHER.

By L. Meunier.

[Condensed from the Collegium.]

The following modification of the method of Nihoul and de Koninck[1] gives good results. A hard glass tube about 50 cm. in length is bent, and drawn out at one end. A plug of glass wool is placed near the bend, next about 20 cm. of pure granulated calcium nitrate (prepared according to directions given by Nihoul and de Koninck), and then a mixture of small fragments of the leather and calcium nitrate. A current of oxygen is led through the open end of the tube, the bent delivery tube dipping beneath the surface of an alkaline solution. The portion of the tube containing calcium nitrate is now carefully heated, and subsequently the heating is extended to that portion containing the leather and nitrate. The leather burns easily in the presence of oxygen, and any volatile products escaping primary combustion are burnt up by the calcium nitrate; the sulphuric acid formed is retained by the lime. When combustion is complete, the receiver containing the alkali is removed, and its contents evaporated to dryness in a platinum dish, incinerated over a spirit lamp, the residue dissolved in hydrochloric acid, and tested for sulphates. If the experiment has been properly carried out, these should be absent. When cool, the contents of the combustion tube are transferred to a beaker with water, and nitric acid neutralized, if present. Hydrochloric acid is then added, and sulphuric acid is determined in the usual way. The process is accurate, but somewhat long and difficult on account of the large quantity of saline matter in the solution.

In the following method, these difficulties are obviated. The leather is burnt in oxygen in a Mahler's bomb calorimeter under a pressure of 30 atmospheres. Four pieces of about 20 mm. diameter and 2 grams in weight, are punched out from different portions of the leather. A similar section is taken from each as an average sample for combustion, the remainder being used for the determination of moisture and normal sulphates, the latter by incineration without alkali. The sample for combustion is mois-

[1] Monit. Scient., 1904, 504.

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tened with a 3 per cent. solution of potassium carbonate, and dried. Fifty cc. of N/10 sodium carbonate solution are introduced into the bomb, and the sample is then placed in the platinum capsule and the bomb closed. The combustion is carried out in the usual way. After completion, the contents of the bomb and capsule are washed out and mixed, and, if necessary, filtered. When combustion has been complete, the filtrate will be colorless. It is acidified with hydrochloric acid, and evaporated to dryness in a porcelain dish. The residue is moistened with strong hydrochloric acid, and re-dried once or twice to expel nitric acid formed during the combustion. It is then dissolved in dilute acid, and the sulphuric acid precipitated as barium sulphate.

If A be the percentage of sulphuric anhydride calculated on the dry leather, B the percentage present in the ash as normal sulphate, and C the percentage corresponding to the average sulphuric acid content of the skin substance of leather, then $X = A - (B - C)$, where X is the percentage of sulphuric anhydride corresponding to the free acid of the leather. C must be experimentally determined once for all.

PATENTS.

U. S. Patent No. 832,786, issued to John Forster, of Leicester, England, covers an improved scouring, setting-out or rolling machine. Present machines for this purpose stretch the leather in one direction at a time, and when the tools change their direction of motion, part of the stretch obtained thereby is the result of the contraction of the leather in the direction in which it was previously stretched. It is claimed for the improved machine that it will give the maximum amount of stretch and flattening in shorter time and with less power. This is effected by causing the rolling tools to operate on the leather in all directions at the same time. The machine is arranged so that three or more tools, or groups of tools, are in action at the same time, and all moving in directions which diverge from the centre of the hide. No part of the leather escapes the action of one or more of the tools. The table carrying the leather may be reciprocated toward and away from the centre of the machine so that all parts of the leather can be conveniently

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rolled. If the claims for this invention are borne out, it should be particularly adapted to rolling "baggy" sole-leather.

U. S. Patent, No. 831,556, issued to Julian Irazoqui, of Caibarien, Cuba, is for an improved vacuum pan adapted to the manufacture of solid sugars or tan extracts. The pan comprises a central chamber, surrounded by a jacket wall forming a chamber for the heating agent, and a plurality of horizontal tubes crossing the central chamber and communicating with the outer chamber. The communicating tubes are arranged within the central chamber in two vertical planes at right angles to each other. A discharge opening is placed at the bottom of the pan and means for admitting compressed air to the central chamber are provided. When evaporation has reached the desired stage, determined by samples taken by means of a closured sampler-tube, heating and vapor pipes are closed, and the extract is forced through the discharge opening by admitting compressed air.

U. S. Patent, No. 834,199, issued to L. H. Francis, of Princeton, California, covers a process for making leather, water and vermin proof and enhancing the wear resisting qualities of the leather. The leather is soaked for from one to six days in a bath of coal tar, heated to 110° F. The leather is then run through press rolls, then dried and finally rolled and glossed.

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G. A. KERR, W. H. TEAS. . . . . Past Presidents

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EDITORIAL.

Four leading English leather chemists have announced that after the first of next January, the purpose to analyze by both the I. A. L. T. C. method and the A. L. C. A. method, and to report the results by each method of analysis. A copy of the announcement appears in this issue aud will be of more than ordinary interest to the members of this Association.

More than passing comment upon this action might be considered impolite and indelicate under the circumstances, and yet no one can quarrel with facts. Two different tannin results by two different methods of analysis, both of which are based on hide powder absorption, would indicate that one method is wrong.

The method which allows of close duplication of results com-

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paring favorably with actual tannery practise, would seem to be the more correct. The dissatisfaction exhibited among the patrons of one method, and the practical unanimity of belief in the other method by those who practise it, admits of but one deduction.

Reporting a tannin analysis by both methods is *per se* a virtual admission of the equality of the two methods; but the subscribers to the agreement go even further than this in their reasons for the proposed action, and practically admit the superiority of the added method and anticipate the permanent adoption of a method based on similar lines. The reputation of the several parties to the agreement is sufficient guarantee of the conservatism and sincerity of the action; as we understand it, the proposition is entirely within the rules of the International organization, and from our point of view seems much more tactful and considerate than the arbitrary action of the German Section. Let us hope that the British characteristic of advanced discounting of anticipations may once more "make good".

THIRD ANNUAL MEETING.

At the 3rd Annual Meeting of the American Leather Chemists' Association, held at the Grand Union Hotel, New York City, November 22nd and 23rd, 1906, the following were present:

H. C. Reed, Stamford, Conn.
Alan A. Claflin, Boston, Mass.
J. W. Phelan, Boston, Mass.
A. L. Dean, New Haven, Conn.
Thos. J. Mosser, Williamsport, Pa.
C. W. Norris, Milwaukee, Wis.
F. W. Ackerman, New York.
F. H. Small, Worcester, Mass.
W. H. McLauchlan, Bradford, Pa.
W. H. Teas, Ridgway, Pa.
A. W. Hoppenstedt, New York.
Charles Eachus, Newark, N. J.
E. J. Haley, New York.
Raymond Greene, Mt. Union, Pa.

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W. A. Fox, New York.
James R. Barbour, Stamford, Conn.
W. H. Bower, Philadelphia, Pa.
W. K. Alsop, New York.
F. Kopecky, Milwaukee, Wis.
F. R. Mosbaugh, Lynchburg, Va.
H. A. Whitman, Damascus, Va.
R. H. Wisdom, Stamford, Conn.
V. C. Casaburi, Wilmington, Del.
E. W. White, Peabody, Mass.
S. Saxe, New York City.
Walter H. Stifel, Allegheny, Pa.
Geo. D. McLaughlin, Buena Vista, Va.
F. P. Veitch, Washington, D. C.
John H. Yocum, Newark, N. J.
Jos. Romadka, Milwaukee, Wis.
Henry W. Ries, New York.
C. R. Oberfell, Newark, N. J.
W. W. Skiddy, Stamford, Conn.

President H. C. Reed in the chair; W. K. Alsop, Secretary. The meeting was called to order at 10:30 A. M., Nov. 22nd and the following program was carried out:—

President's Address.
Secretary and Treasurer's Reports.
Report of Business Manager of JOURNAL.
Referee's Report.

2ND SESSION 2 P. M.

New By-laws were introduced and adopted.

Paper by F. W. Alden, "Moisture in Sod Oil and Moellon."

Paper by W. H. McLauchlan, "Determination of Volatile Acids in Commercial Lactic Acid."

3RD SESSION, NOV. 23RD, A. M.

Paper by Ferd. Kopecky, "Analysis of Dyewood Extracts."

F. P. Veitch, "Chromed Hide Powder—Extemporaneous."

H. C. Reed, "A Comparison of Chromed Hide Powders."

S. C. Dodge, "A New Method for the Analysis of Tanning Materials."

Secretary read a resolution expressive of regret at the death of W. H. Krug.

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A letter from I. A. L. T. C. was read and the Secretary was instructed to reply.

H. C. Reed read his report to the A. O. A. C. in capacity of their Referee on Tannin.

H. C. Reed read such parts of A. O. A. C. Provisional Method as differed from ours.

Vote of thanks extended to the Referee.

4TH SESSION, 23RD, P. M.

Committee on Methods for Sampling reported and the Association adopted Provisional Method.

F. Kopecky, paper "Analysis of Alkaline (Tannin) Solutions."

J. H. Yocum, paper, "Leather Analysis."

Election of officers, to serve from Jan. 1st, then took place with the following result:

President, J. H. Yocum; Vice-Pres., F. H. Small; Sec.-Treas., W. K. Alsop.

Ordinary Members of Council: One year, F. R. Mosbaugh, E. J. Haley. Two years, H. C. Reed, H. T. Wilson.

ANNOUNCEMENT.

To the Editor of the "*Leather Trades Review.*"

DEAR SIR:—At the recent Conference of the International Association Leather Trades Chemists, held at Frankfort at the end of September, a long discussion took place with regard to the official method for the analysis of tanning materials and extracts, and from various sides, notably from the English and American members of the Conference, great dissatisfaction was expressed with our present method, owing to the increasing difficulty of getting a uniform hide-powder. We are aware that this dissatisfaction is shared by the leather trade in general in this country, but we have been averse to altering the method until we could be sure that we were making a permanent change for the better. Early in the present year the German Section of the International Association changed from the official hide-powder to a specially prepared chromed hide powder, and are at present using this hide-powder for all their analyses, but stating the fact on their reports, as is permissible by the rules of the I. A. L. T. C. We in this country are not convinced that this chromed hide-powder is more accurate than our official method, since the analytical re-

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sults give on an average at least 1% higher in tannin. This action of the German Section has lead to great inconvenience and misunderstanding, through the German, French, and Austrian extract manufacturers selling on the German analysis, and the English buyer stipulating for the official, according to his contract. The change in hide-powder made by the German Section was adopted after mature consideration and for sufficient reasons, and as at the Frankfort Conference it was found impossible to come to an immediate decision, an International Commission, consisting of one leading chemist from each country, with Professor Procter as chairman, was appointed to draw up a standard method of analysis, which if approved of by the Executive Committee will come into force for all sections of the I. A. L. T. C. on June 1st, 1907. As the American leather chemists in their excellent journal have published a mass of results by their method of analysis, showing a uniformity and accuracy which it is impossible to obtain by our method, we feel that the new method of analysis will be based on similar lines to the American, especially as this is a similar method to that now adopted at the Vienna Research Station by Director Fitner. Moreover, it has been proved that by the American method a faulty hide-powder can be treated and made quite equal to the best hide-powder by light chroming and washing. One of us has recently carried out a series of of analyses with eight different powders, six of which were condemned as inaccurate and useless for our present method while the other two powders were of first-class quality, and were passed by the hide-powder committee. The eight analyses agreed one with another within two-tenths per cent. Further, it has been shown that the American shake method does not absorb glucose, dextrine, gallic acid and other non-tannin compounds frequently present in extracts, to nearly the same extent as our present method, by which considerable quantities are necessarily reported as tanning matters.

On these and other grounds we purpose from the 1st of January onwards reporting all analyses in duplicate, giving in one column the analysis by the standard I. A. L. T. C. method, and in the second column, for the sake of comparison, the figures obtained by the standard American method. By doing this we trust that the trade will gradually become accustomed to the change which we think is likely to take place in June of next

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year, and by making this public, we hope to correct the many inaccurate statements that are now being circulated throughout the leather trade with regard to altered methods in analysis, faulty hide-powder, dissension among the members of the I. A. L. T. C., etc., etc., all of which is devoid of foundation. Tanners, in making contracts for extracts or tanning materials for the next year, would be well advised to have their analyses carried out in duplicate by the two methods, for which extra work we purpose for the present to make no additional charge.

We are, dear Sir,

Yours faithfully,

(Signed) ROBERT HELLON,
J. GORDON PARKER,
H. R. PROCTER,
ANDREW TURNBULL.

REPORT OF THE REFEREE, 1906.

The ground to be covered by the research work of the Association for the current year was outlined in part by last year's Referee in his list of recommendations and further defined by the general sentiment of the last Annual Meeting that the methods of Tannin Analysis of the Association were now sufficiently satisfactory to render it desirable to make a start on the formulation of Official Methods of analysis of other tannery materials. In appointing the several research Committees and outlining for them the subjects for investigation, the Referee, therefore, attempted to keep within the rather wide limits so suggested. The continuance of the work outlined by his predecessor in office seemed of the first importance and after providing for this the Referee found that the men available for work in new fields were so few he would be compelled to keep pretty much to the well beaten paths. A small start afield was made which he hopes another year may see greatly increased.

The subjects for research recommended by last year's Referee were as follows (See Journal 1-1, p. 116.)

1. Soluble solids filtration with especial reference to the time-contact method so termed in conjunction with the temperature problem.

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2. Analysis of liquors and the effect of the acidity of liquors upon the analysis.

3. Estimation of acid in tan liquors with a view of improving the present method.

4. The influence of acidity and alkalinity upon the chroming of hide powder and upon the subsequent analysis. The elimination of the sulphate factor and the merits of a fully or partially chromed prepared hide powder.

5. Extraction, for the purpose of confirming or rejecting the recommendations of the Referee.

6. The Parker-Payne method of tanning analysis.

7. Estimation of nitrogen in leather and tan liquors.

Under the head of Color Tests he recommended that the entire matter of color determination be made a subject of investigation next year and that in addition to the lines of work indicated, the usefulness of the Tintometer for the purpose be investigated.

The Association referred to the Referee the matter of drawing up a method of sampling, instructing him to appoint a Committee to study this subject and report.

The following committees were appointed :

1. Soluble Solids.—Reed, Ch., Mosbaugh, Nichols, Evans, McLaughlin.

2. Chroming of Hide Powder.—Norris, Ch., Alden, Teas, Ackermann, Drueding.

3. Extraction.—Alsop, Ch., Veitch, Russell, Mosser, Reed.

4. Liquor Analysis.—Cushing, Ch., Fox, Wilson, Drueding, Mosbaugh.

5. Acid in Liquors.—Hoppenstedt, Ch., Eachus, Evans, Nichols.

6. Color and Identification of Tanning Materials.—Veitch, Ch., Kerr, Loveland, Stillwell, Small.

7. Leather Analysis.—Yocum, Ch., Mosser, Casaburi, Ackermann.

8. New Methods.—Fox, Ch., Claflin, Casaburi, Holmes.

9. Commercial Acids.—Teas, Ch., Claflin, Eachus, Alden, Russell.

10. Sampling.—Haley, Ch., Yocum, Kerr, Cushing, Norris.

Committee No. 7 was asked to draw up a method for the Analysis of leather : to determine which form of the Kjeldahl Method is most satisfactory for the estimation of Nitrogen in

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leather, tan liquors, etc., and whether the method fails in the presence of chlorides. (Collegium No. 158, pps. 167–168.) This Committee unfortunately rendered no report.

Committee No. 8 was asked to test experimentally and report on the value and utility of the various new methods that have been and are proposed for the estimation of tannin.

The Referee could find no one willing and able to accept the Chairmanship of this Committee and it consequently has no report. This is the less to be regretted as the particular method the study of which was recommended by last year's Referee, namely the Parker-Payne Method has been conclusively shown (by the researches of Procter and Bennett (J. S. C. I. XXV No. 6, p. 251)), to be unreliable. No other methods have been published which seem likely to be of value for the immediate purposes of this Association unless the Sprouted Alumina method of Wislicenus may be an exception. The usefulness of this method is certainly far from demonstrated.

Committee No. 10 was asked to draw up a method for sampling the various tanning materials in any quantity and in any kind of package.

The Referee has received a copy of a provisional outline of such a method which he understands Mr. Haley, Chairman, expects to present for the consideration of the Association.

Committee No. 3 was asked to investigate the following questions: Does prolonged boiling of an extraction liquor alter its tannin content? Does the layer of cotton called for by the Official Method absorb any tannin that is not later returned to solution? When a percolator is used is it necessary to heat the percolate to 80° C. to insure solution of all soluble matter? Is it desirable in all cases to extract such an amount of material as will give a liquor of Official strength for analysis? If the ratio of Hide Powder to Total Solids is constant does the concentration at which the liquor is analyzed affect the tannin figure? What conditions are necessary to secure the maximum percentage of tannin in extracting new and spent hemlock, oak bark, and chestnut wood? Extend this investigation to other materials if time permits.

The Chairman of this Committee sent the Referee a provisional report of work in progress but was unable to place any definite results in the hands of the Referee in time to assist him in laying

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out the general work. The Referee, therefore, thought it wisest to omit this subject from the general work and to attempt no discussion of the several questions above enumerated.

Committee No. 6 was asked to "compare the Lovibond Tintometer, the skiver method and the use of mordants on cloth as to the reliability and accuracy of the information they give with regard to the color values of tanning materials. Study the reactions of the common vegetable tanning materials with a view to establishing a method for the qualitative identification and quantitative estimation of each in presence of one or more of the others."

The second subject was not touched upon by the Committee. It presents great difficulties but its importance warrants a large expenditure of time and labor and the Referee hopes some future Committee may be able to give to this question the attention it deserves. It was hoped that the color work with mordanted cotton might bear on this subject, but the Chairman reports "Slight differences in strength of solution give differences in color and I am inclined to think that it will be difficult to duplicate results. I do not feel that one would care to say that an extract was adulterated say a Sumac with Lentiscus or an Oak with Chestnut, Hemlock with Quebracho, etc., from the colors produced on cotton." The usefulness of mordanted cotton for this purpose has been studied rather fully by Dr. Paessler who presented a paper on this subject at the recent conference of the I. A. L. T. C. in Frankfort. It was the general feeling of those present, however, that no certainty of differentiation was possible by the method.

The Referee would call attention to the conclusion of the Chairman concerning the usefulness of the Lovibond Tintometer: "It would seem, therefore, that while the tintometer will serve a very useful purpose in checking up the deliveries of a given extract and also in manufacturing extracts it will still be necessary to make a color test on hide when it is desired to determine the colors given by the different kinds of extracts." The method would seem, therefore, of so limited applicability that the Referee doubts the wisdom of asking the Association to devote further time to its consideration.

The best method of making color tests with hide is discussed at some length in the report. The Referee considers the recommenda-

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tion of the Chairman (Journal 1, 8, pps. 260–261), in general sound except that he feels doubtful of the development of a full color in the short time suggested for contact of hide and liquor. Until a larger study and comparison has been made in Committee of the various factors which influence the color, so that details may be intelligently prescribed, the Referee feels unwilling to offer a Method for the consideration of the Association. He feels that a continuation of the work both with mordanted cotton and hide skivers is desirable.

Committee No. 9 was asked to work out a method for the estimation of the percentage of acid in the ordinary commercial acids used by tanners, such as Lactic, Formic, Pyroligneous, etc.

Mr. Teas, Chairman, presented a report for the Committee, to be found in the Journal, 1, 5, pp. 214–217. The results reported seem to make it needful to emphasize the absolute necessity of correct standard solutions. There is a constant variation in the results reported by the different members of the Committee which undoubtedly is due to a variation in the standards employed. The method for the determination of volatile acids in Lactic Acid which was tested by the Committee gave very concordant results, but no proof was brought forward that the results obtained represented the actual percentage of volatile acid or that the agreement was other than a concordance to be expected from any empirical process. The work of the Committee in general was not carried far enough so that the Chairman felt willing to recommend any definite methods, his final summary being, "As this is the first work the Association has undertaken in this line the Chairman feels neither qualified nor at liberty to make any definite recommendations as to methods." The Referee felt no more competent than the Chairman to make such recommendations, and as he believes the function of the general work is to test definitely-outlined methods rather than to work out details of procedure, he felt obliged to omit any work on this subject from the general collaboration. The Referee believes a good start has been made, and that the work in Committee should be continued until such time as the Committee is ready to recommend to the Association for trial, methods which it considers satisfactory and accurate.

Committee No. 5 was asked to compare the Official Method,—the Procter Method,—the Gelatine-Precipitation Method, using

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a slightly alkaline brownish turmeric paper as indicator,—and any other method that seems practicable, (Leather Manufacturer, Dec. 1905, p. 220,) for the determination of the acid in tan liquors.

The report for this Committee by Hoppenstedt, Chairman (Journal 1, 6, pp. 221–228) is perhaps the most valuable recent contribution to this subject. It indicates that practically all of the methods which have been proposed for the estimation of the acidity of tan liquors fail to show the full amount of acid contained, and further fail to give good comparative results. A new method whereby the tannin is precipitated from the liquor with Quinine, the liquor filtered, and the Quinine salts of the free acids originally present, titrated in the filtrate with alkali, phenolphthalein being used as indicator, was proposed by the Chairman and in the hands of the Committee yielded excellent results. The Referee had great hopes that the difficult problem of the accurate determination of the free acid in tan liquors was at last satisfactorily solved. When he tested the method, however, he found that with certain classes of liquor it failed to work. The trouble arose from the fact that the Quinine solution to be titrated, darkened so rapidly that it became impossible to get an end point with the indicator. Various methods of manipulation were tried, but without successful results. Mr. Hoppenstedt wrote the Referee in this connection "I have been looking into the matter and have come to the conclusion that it must be the gallic acid which causes the trouble as it only appears in some oak liquors. I have further found that this darkening is prevented to a great extent by adding some C. P. zinc oxide after precipitating the Quinine solution, stirring well, and then filtering. Apparently zinc gallate is formed and removed." The Referee acted upon this suggestion, but found that unless the zinc oxide remained in contact with the liquor for from eight to sixteen hours it failed to improve matters much. Long contact made it possible to obtain a filtrate with which a satisfactory end point could be secured. Under such conditions, however, it seemed probable that a considerable change from the original acidity of the liquor might take place. The method so amended further involves considerable delay. After consultation with Mr. Hoppenstedt it seemed wisest to allow him a longer time in which to perfect his method before submitting it to the Association for general test.

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Committee No. 1 was asked to compare the present Official and Experimental methods of determining soluble solids:—to determine whether a Centrifuge under similar conditions separates out a constant amount of residue:—to decide whether there is any advantage in using a strong liquor, say 10° Bk. for the Insolubles determination.

Mr. Reed, Chairman, presented a report for the Committee, to be found in the Journal, **1**, 7, pp. 242–247. No work was done with the centrifuge. The results with a 10° Bk. liquor were not encouraging, the variation in the Insolubles figures being relatively large. The Chairman remarks that "Since it is admitted that analyses at different dilutions will give different results it is far preferable to use a dilution that is capable of returning figures in concordance rather than to adopt a dilution that will give results similar to those obtained under Study 2."—that is using a 10° Bk. liquor for the Soluble Solids determination. This conclusion of the Chairman seems to the Referee sound and he sees no reason to recommend any change in the method along this line. The only valid reason for using a stronger dilution than at present for the soluble solids determination would be to secure a closer agreement of analysis figures. It is manifestly impossible to determine for the tanner the amount of insoluble matter that will actually settle out from liquors made from the extracts, analysis of which is made for him. The amount depends primarily on the concentration, and in liquors of the same concentration is variable and dependent upon many conditions: in new liquors especially it is not a constant but increases with age. Finally much of the insoluble matter which settles out is not really insoluble but merely difficultly soluble Tannin, which ultimately combines with the hide and helps in the leather-forming process.

The results of the comparison of the Official and Experimental methods for determining Soluble Solids in the case of a chestnut wood, a quebracho and a hemlock extract were rather in favor of the Official Method as far as concordance goes, but Mr. Reed remarks that "This may be due in part to lack of acquaintance with the manipulation of the time contact method," and "recommends that the time-contact method be given a trial in the general collaboration."

This was accordingly done and made up Study II under Extracts, Study I being an analysis by the present Official Method.

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A further basis of comparison was furnished by the collaborative work on the liquor samples.

Directions for Study II were as follows: To 1 gm. of kaolin in a beaker add 75 c. c. of solution; stir and pour on No. 590 S. and S. 15 cm. pleated filter paper; return filtrate to paper for one hour, keeping filter full. At the end of an hour, pour solution from filter or remove with pipette. Bring 800 c. c. of solution to 20° C.; refill the filter with this solution and begin to collect filtrate for evaporating and drying so soon as filtrate comes CLEAR. Keep filter full. Note:—all pipettings are to be done at room temperature, which should be held as nearly constant as possible throughout the work and between 20° and 25° C.

In Studies IV and VI on Samples D. and E., the Official Method of Soluble Solids determination and that outlined under study II were respectively used, so that while the fact that the dilutions of the liquors were not the same in the two studies makes a direct comparison impossible, the degree of variation in the results obtained in the two studies is helpful in a valuation of the two methods.

The figures will be found in the accompanying tables. The best that can be said of them is that they are not very conclusive. The Insolubles by the experimental method are in general rather higher than by the Official Method, but the filtrates are reported brighter and clearer. The difference is small at best. With samples A and C the widest variation occurs with the Official Method,—with B by the experimental. The same lack of decisive superiority of one method over the other is found in the liquor work. With sample D, a liquor low in tannin and purity, the two methods give practically identical results; with E, a liquor of higher tannin content and higher purity the Official Method shows a slight superiority. It seems in general true that in all these cases if we adopt a certain arbitrary limit either side of the average, representing what might be assumed to be a normal limit of accuracy,—say 10% of the total,—a larger number of results will be found outside these limits in the case of the Official than in the case of the Experimental method. Applying this test we find for the different samples results outside the limits as follows:—

Sample	A	B	C	D	E
Official	5	17	6	6	7
Experimental	5	15	3	5	6

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The results show further an undoubted saving of time by the Eperimental method in most cases. Moreover the conditions were favorable for the Official Method in that the temperature at which the work was conducted by the different collaborators was remarkably constant and that the collaborators were more familiar with the Official than with the Experimental Method. The Experimental method would seem to have one further advantage over the Official in that the time of contact of liquor and filter paper being more nearly constant, the loss by absorption in the case of extracts of different kinds would be rendered more nearly equal. By neither method is the absorptive power of the filter paper and kaolin fully satisfied. It may well be that part of the variation is due to the filter paper used, for S. and S. No. 590 filter paper has been poorer in quality this year than at any previous time in the writer's experience. It would seem very desirable to have an alternative paper to fall back upon in such a contingency. The Referee has found Dreverhoff No. 400 a satisfactory paper and would recommend that it be given a trial with a view to making its use for Soluble Solids determination official.

Feeling that the advantages are with the Experimental Method, and that a greater familiarity with the method will result in a more decisive showing of superiority of the same, the Referee recommends that the Experimental Method as laid down in Study II be made the Official Method for the determination of Insolubles.

Committee No. 2 was asked to study the following subjects—Comparison of partially chromed and fully chromed hide powder, and hide powder manufactured according to the method proposed by Kopecky. The best method of chroming hide powder and the best compound of Chromium to use. If the present method be used are soluble salts or soluble hide introduced into the non-tannin filtrate? Will the use of a trace of barium chloride in the first wash waters shorten the time of washing, or affect the solubility of the chromed hide powder? What effect does the condition of the hide powder as to alkalinity or acidity have on the amount and rate of absorption of tannin?

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a. In extract or sweet liquors.

b. In sour liquors.

If the concentration of the analysis solution and the quantity of hide powder used are varied in the same proportion, is the tannin figure obtained the same?

The amount of work assigned to this Committee was rather large, and it is not to be wondered at that Mr. Norris, the Chairman, reported that "The Committee * * * found it impossible to carry out all the work outlined by the Referee." Full details of the, by no means inconsiderable, work done by the Committee may be found in the Chairman's report (Journal 1-5-pps. 203-208.)

The first question submitted to the Committee was gone into rather fully. Hide powders prepared both by Mr. Teas and Mr. Norris after the formula of Dr. Kopecky were tried on a quebracho, a hemlock and a chestnut extract. Non-tannin figures agreeing fairly well among themselves were obtained, as shown in the report. Mr. Norris later forwarded the Referee a sample of Dr. Kopecky's hide powder furnished by Dr. Kopecky himself, which because of insufficient size to make it possible to use for the general work, the Referee tried to test out himself. The hide powder is rather difficult to soak up with cold water, but by using hot water as recommended by Dr. Kopecky is readily soaked and washed and very easily squeezed to the Official water content: tested for acidity according to the method suggested by the Referee, it required 5.8 c. c. N/10 alkali. Using this hide powder on the Official samples and according to the Official Method, values for Non-Tannin were obtained as in the table below. The extract filtrates were all colored, A and C very highly,- and all gave a prononnced test with a gelatine-salt solution. The Non-Tannin filtrates from D were clear and nearly colorless but from E were colored and at the Official dilution gave a strong test with gelatine-salt. Thinking that longer contact of hide powder and liquor as recommended by Dr. Kopecky might improve matters, the hide powder and liquor were macerated two hours and then shaken ten minutes with results as per table. The filtrates were less highly colored though C was still distinctly red. A and B gave doubtful tests with gelatine-salt solution while C still gave a strong test. The filtrates were in general difficult to filter clear.

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COMPARISON OF OFFICIAL AND KOPECKY HIDE POWDER.

Sample Number	A.	B.	C.	D. IV.	D. VI.	E. IV.	E. VI.
Official H. P.....	10.91	10.75	14.11	5.67	5.49	7.09	6.67
Kopecky H.P., Off. method.	19.80	13.29	20.81	5.61	5.35	7.38	6.81
Kopecky H.P., macerated 2 hrs., shaken 10 min....	14.02	10.97	17.95	..	..	..	6.35

These results would be more conclusive if corroborated by further experimental evidence, but they are in line with the results secured by Mr. Reed last year, and indicate that nothing is to be gained by the use of the Kopecky hide powder. It cannot be used successfully by the Official Method. It necessitates long contact of hide powder and liquor, thus making the value obtained dependent upon the time of contact and so introducing a needless source of variation, or it necessitates the acidification of the analysis solution, an equally needless complication.

In this connection the Referee would state that he received from Mr. Alden a sample of dry chromed hide powder, to be used without previous washing. This was tested by the Referee, but with results of little promise. The hide powder, when used, was extremely non-absorptive and difficult to wet. The following values for Non-Tannins on the Official samples were obtained by macerating the hide power and liquor two and one-half hours, with frequent shaking.

Sample	A.	B.	C.	D. VI.	E. VI.
Alden H.P., Non-tannin....	14.65	11.33	18.61	5.67	7.27

These filtrates, with the exception of D., were colored, and gave a test for tannin with gelatine-salt. In reference to the results, Mr. Alden writes: "I see one possible cause, and perhaps two, why you do not get good results. In the first place, I intended to write you, giving my full directions as to the use of the hide powder. In my work with this powder I used 16 gms. for each determination, since considerable foreign matter has been added to the powder, but this difference, perhaps, would not alone account for poor results. I do think, however, that where the difficulty lies is that on long standing this hide powder loses its absorptive power, either through some deterioration (hardening) of the hide substance, or loss of moisture, thus rendering it less readily soaked up. I have already observed that this dry hide powder has a tendency to lose its absorptive power, and so do not feel very enthusiastic on the dry hide powder question. This

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same powder, when freshly prepared, gave results agreeing quite closely with the Official Method, both by myself and Mr. Eachus." The experience of the Referee and the conclusion of Mr. Alden are fully corroborative of last year's work and the judgment of Mr. Reed.

Mr. Alden has further suggested the desirability of chroming in a 5% salt solution when a large amount of basic chrome salt is used. He writes: "When a salt bath is not used, the leather produced is hard and tender, for the reason that the chromium seems to injure the hide substance when a hydrolizing agent is not present * * *. Chroming the hide powder in a salt solution increases its absorptive power to quite a marked extent." In support of this Mr. Alden has sent results on the Official extract samples as follows:

SAMPLE	A.	B.	C.
Chromed with salt	9.75	9.95	12.29
Chromed without salt.....	10.23	10.15	13.10

Returning now to the question of lightly and heavily chromed hide powder, Mr. Norris's conclusion is that "The absorption of the hide powder decreases with heavier chroming, while there is an increase of absorption with the increasing basic property of the liquor." This conclusion is hardly borne out by the Committee results, and the variation in the results obtained by chroming with varying amounts of chrome solution of varying basicities would seem to fall most largely under the head of personal error, so little do they seem to follow any law. To throw some light on this question, if possible, Studies I. and III., IV. and V., VI. and VII. were put in the general work. Studies I., IV. and VI. call for Non-tannin determinations by the Official Method, with officially-chromed hide powder. The directions for Studies III., V. and VII. were as follows:

Determine Non-tannins as by the Official Method, except use hide powder chromed as follows:—Dissolve 100 gms. of chrome alum in water; add 12 gms. of dry sodium carbonate in solution and make up the whole to one liter. Digest the required amount of hide powder in 25 times its weight of water and add 1.5 cc. of the above solution for every gram of hide powder taken; stir occasionally and let stand 24 hours; wash free from sulphates as usual and press to contain 70–75% moisture.

The results from this work will be found in the accompanying

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tables. They are as little decisive as were those on the Soluble Solids work. In order to eliminate variations due to drying and Soluble Solids determination, a figure obtained by subtracting the Non-tannin value from the Total Solids is best used. For convenience of comparison, the averages of this figure for the several samples are here tabulated :

SAMPLE	A.	B.	C.	D.IV.	D.VI.	E.IV.	E.VI.
Off. Chromed H.P.....	76.31	35.14	32.63	.96	1.13	4.61	4.93
Hvy. " "	76.06	35.12	32.49	.99	1.18	4.65	4.92

The difference between the results with the two hide powders is in general well within the limit of experimental error. A careful comparison of the full tables will show very little in favor of either as regards concordance of results, the one excelling the other about an equal number of times. Applying the same test as when discussing the Soluble Solids results, and allowing a variation of 1% for extracts and 5% for liquors, the results outside these limits are as follows :

SAMPLE	A	B	C	D. Iv	D. VI	E.IV	E.VI
Off. Chromed H. P.....	7	7	8	11	8	4	4
Hvy. " "	5	4	11	9	6	5	6

It would seem, therefore, that so far as concordance is concerned it is immaterial whether a lightly or heavily chromed hide powder be used. As regards ease of handling, the heavily chromed hide powder has the preference. It is very readily squeezed to the proper moisture content and breaks up into a beautifully fluffy mass—neither of which remarks can be made of some of the excessively acid hide powder which has been on the market of late. As opposed to this, the heavily chromed hide powder is much more difficult to wash free from sulphates. Believing that it is desirable to limit the acidity of the hide powder used for analysis, which if done will take care of the question of ease of manipulation, the Referee sees no sufficient reason for recommending a change from the present method of chroming of hide powder.

Questions 2, 3 and 4, namely, the best method of chroming,—the solubility of the hide powder,—the use of barium chloride in washing,—assigned to the Committee have received no attention so far as the Referee knows. To throw some light on question 5, namely the effect of the proportion of acid in the hide powder, the Referee asked the collaborators to determine the acidity of the hide powder used by them as follows—

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Digest 5 gms. of the hide powder in 75 c.c. of water for 2 hours; add 5 drops of phenolphthalein and titrate with N/10 alkali until the addition of two drops of alkali gives a pink coloration lasting for one minute. Report the number of c.c. of N/10 alkali required.

This method was not worked out with special thoroughness by the Referee, and both Messrs. Cushing and Veitch have called his attention to the fact that better results would probably have been obtained had some other indicator—such as azolitmin —been used. This being granted and the desirability of a more careful prescription of modifying details conceded should some such test for a standard hide powder be thought necessary, it will doubtless be granted that the results secured by the above outlined test should form a reasonably satisfactory basis for a comparison of the acidity of the different hide powders employed and thus of the results to be expected from hide powders of varying acidity.

The hide powders employed by the participants in the general work may be divided into three groups as follows—

Group I	N/10 Alk.	0.0— 5.5 cc.	6	collaborators
" II	"	8.2—10.0	5	"
" III	"	10.7—14.1	7	"

Using again as a basis for comparison the difference between the Non Tannin and the Total Solids figures we have in the average.

OFFICIAL HIDE POWDER

SAMPLE	A	B	C	D.IV	D.VI	E.IV	E.VI
I	76.39	34.70	32.42	1.02	1.13	4.65	5.01
II	76.11	35.17	32.79	.89	1.10	4.56	4.85
III	76.39	35.50	32.56	.93	1.13	4.63	4.90
		HEAVILY CHROMED HIDE POWDER					
I	76.78	35.08	32.54	1.01	1.19	4.72	5.02
II	75.46	35.04	32.50	.90	1.13	4.56	4.83
III	76.00	35.19	32.44	.98	1.21	4.67	4.90

Here again the purely personal factor seems to cause enough variation to obscure any variation due to the acidity of the hide powder. The hide powder of medium acidity in the table gives a consistently lower tannin figure than the very neutral or very acid powder, but a study of the components of this average shows that the variation in these several components is far more than between the averages of Group II and Groups I and III. In short so far as these results go we may conclude it is imma-

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terial what the acidity be of the hide powder used. But if we grant this as to results (and the Referee is not willing to grant it for various reasons some of which will be mentioned later on) it certainly is not immaterial as regards ease of manipulation. It is almost impossible to squeeze some of this highly acid hide powder to the Official moisture content, 70—75%, and when so squeezed it is practically impossible in any reasonable time to reduce it to a condition sufficiently fluffy so that when carefully examined after having been used for detannizing it will not show numerous kernels with centers not even colored by the tannin solution; in other words a distinctly less amount of hide powder than that weighed out will have actually been active in the process. The Referee, therefore, believes that limiting values for the acidity of hide powder acceptable for analysis should be prescribed in the method and he recommends that the most suitable limits be worked out by a Committee and made a part of the requirements of the Official Method.

In this connection Mr. Veitch writes "The hide powder used was Vienna and was a very unsatisfactory lot, being hard and flinty and having an alkaline reaction. It contained 12.37% moisture and 5 gms. digested with 75 c.c. of water and titrated required 4 c.c. N/10 alkali to make alkaline to phenolphthalein, but required 10 c.c. of N/10 acid to make acid to methyl orange or litmus. I do not think that the results of this titration, using phenolphthalein as indicator are accurate as an alkaline hide may appear faintly acid. Litmus would probably be better. After chroming by the Official Method the hide was still faintly alkaline and slow in absorbing tannin. The results, therefore, with the extracts are far from satisfactory, as the non-tannin residues in nearly all cases contained a little tannin, filtererd very slowly and were inclined to be turbid. The use of more hide or longer shaking would probably have given better results. The figures, however, are of value in showing the unsatisfactory working of a poor hide. On the extracts I have included results with the same hide made acid with H_2SO_4 and washed free of sulphates. The filtrates were free of tannin, were bright and filtered readily. When the hide was heavily chromed it did better work and in all cases worked well with liquors. * * * If heavy chroming is going to give lower Non-tannins than our present method (and it appears that it will) I am not in favor of it."

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Before passing on, the Referee wishes to call attention to the exceeding carelessness of the collaborators in keeping the amount of hide powder used, within the required limits. Amounts of dry hide powder ranging from 8.4 gms. to 15.4 gms. were used, the variation being sufficient in many cases to effectually mask any change due to methods of chroming, percentage of acid, etc. Very few adhered rigidly to the requirements of the method and kept the amount of hide powder used within the limits, that is between 12 and 13 gms.

Question 6 on the effect of varying the strength of the analysis solution and the amount of hide powder used proportionally was assigned the Committee in the hope primarily of throwing some light on the question of the analysis of extraction liquors from spent materials which naturally run off less than Official concentration. No results were returned by the Committee. A few results obtained by one of the Referee's assistants may be of interest and are here recorded.

Kind	Gms. per liter	Tot. Sol.	Sol. Sol.	Non Tan.	Tan.	Ins.	Gms. Dry H. P.
Quebracho	5.9946	87.51	79.91	10.64	69.27	7.60	12.46
"	2.9982	88.39	83.05	13.46	69.59	5.34	6.23
"	1.5084	90.96	86.58	15.52	71.06	4.38	3.12
Chestnut	14.9752	38.48	38.29	12.73	25.56	.19	12.46
"	7.5522	38.64	38.27	14.50	23.77	.37	6.23
"	3.7380	38.79	38.47	16.61	21.86	.32	3.12
Hemlock	14.0130	46.83	43.27	13.53	29.74	3.56	12.46
"	6.9998	47.00	45.54	16.11	29.43	1.46	6.23
"	3.5198	47.22	46.99	17.92	29.07	.23	3.12
Mangrove	12.6334	47.49	43.79	13.16	30.63	3.70	12.66
"	6.3178	48.18	46.76	14.88	31.88	1.42	6.33
"	3.1598	48.93	48.42	16.61	31.81	.51	3.16
Chestnut	15.0360	37.60	37.42	10.32	27.10	.18	12.82
"	7.5254	37.55	37.42	11.60	25.82	.13	6.41
"	3.7570	37.53	37.74	13.60	24.14	..	3.20
Hemlock	14.1576	43.85	41.21	11.84	29.37	2.64	12.82
"	7.0804	43.92	42.54	13.93	28.61	1.38	6.41
"	3.5354	44.29	44.35	15.64	28.71	..	3.20

From these results it would seem that the non tannin figure decreases as the concentration of the analysis solution increases and that the soluble solids figure decreases likewise. A partial compensation therefore, results as regards the tannin figure which, however, varies with the different materials.

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Committee No. 4 was asked to investigate the following matters—

Compare the present Official and Experimental methods of liquor analysis.

Suggest any desirable modification of whichever of the above appears the better.

What effect do the rate of shaking and the time of shaking have on the amount of matter absorbed from a liquor by the hide powder?

What difference results from diluting with water at 80° C. or with water at room temperature, more particularly in the case of fairly strong and fairly new liquors?

What effect does the acid in the liquor have on the analysis figures?

Mr. Cushing for the Committee presented a most complete and interesting report to be found in the Journal 1, 6, pp. 228-239. Taking up the above matters in reverse order, the report of the Committee with regard to the effect of the acid in a liquor on the analysis figures is rather contradictory to the report of last year's Committee, but neither report pretends to be at all exhaustive and the Referee is content to acquiesce in the judgment of the Chairman, Mr. Cushing. "It must be left to some future Committee to more fully investigate the effect of acids upon the analysis figures. From indications it should take an important place in future investigations."

As regards the effect of diluting with water at 80° or at room temperature the results of the Committee would indicate that the hot dilution causes a slight solution of Insolubles, thereby lowering the Insolubles figure and increasing the Tannin figure. No effect on the Non-tannin figure is apparent. In general the variation is small.

To get some further light on this question the Referee had Insolubles determinations made on several liquors at full strength and when diluted to analytical strength with water at room temperature and with water at 80° C.

Results were as follows:—

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	Bk.	T. S.	S. S.	Ins.	24° C. T. S.	S. S.	Ins.	80° C. T. S.	S. S.	Ins.
Dilution		1000–1000			90–1000			90–1000		
Hemlock.....	30	7.45	7.34		7.39	7.27		7.38	7.35	
		7.45	7.38	.09	7.38	7.27	.12	7.40	7.36	.03
Dilution					600–1000			600–1000		
Hemlock.....	15	3.34	3.25		3.34	3.23		3.33	3.23	
		3.35	3.25	.10	3.33	3.25	.10	3.33	3.22	.10
Dilution					60–1000			60–1000		
Oak	51	13.02	12.84		12.99	12.70		12.94	12.77	
		13.02	12.88	.16	12.95	12.71	.26	12.96	12.76	.18
Dilution					70–1000			70–1000		
Oak	52	12.08	11.92		12.16	11.79		12.13	11.88	
		12.08	11.93	.15	12.15	11.81	.36	12.16	11.88	.27
Dilution					400–1000			400–1000		
Oak	31	7.53			7.46	7.35		7.49	7.38	
		7.51	7.40	.12	7.48	7.37	.11	7.51	7.42	.10
Dilution					800–1000			800–1000		
Oak	27	4.32	4.22		4.33	4.22		4.31	4.20	
		4.32	4.23	.10	4.33	4.21	.11	4.32	4.22	.11

These figures would seem to suggest that neither method of dilution shows the amount of Insolubles that is present in the original liquor, a hot dilution tending towards a lower Insolubles figure, a cool dilution toward a higher. Apart from bringing the method for liquor analysis in line with that for extracts the only object in a hot dilution would seem to be to attain greater concordance and as the Committee results fail to show a gain in this direction, the extra labor involved in the hot dilution would seem uncalled for, and it was consequently omitted from the experimental method as outlined for the general work.

The results of the Committee seem conclusive as to the non-effect of the rate of shaking on the analysis figures, so long, as Mr. Cushing says, "As the speed is sufficient to properly agitate the mixture of hide and liquor."

Mr. Mosbaugh in commenting on the general work, says, however, "In comparing results, especially those obtained from five minute shaking, I find that there is not such a decided difference as our Committee doing this same work found last year. I at-

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tribute this to an increased speed of my shaker as in my opinion the amount of agitation given is an important factor and should be regulated by the association."

With regard to the time of shaking, Mr. Cushing, in his report, says "Study E is interesting as an indication that a reduction of the time of shaking is possible. The writer recommends that this point shall be made a subject of investigation in the collaborative work of the members of the Association this year."

This recommendation was carried out by the Referee and tested on both extracts and liquors with both Official and heavily chromed hide powder. The work was contained in studies, Ia, IIIa, IVa, Va, VIa, and VIIa, the directions for which called for non-tannin determinations under precisely the same conditions as studies, I, III, IV, V, VI and VII respectively except that the time of shaking in Ia, etc., was 5 minutes instead of the 10 minutes in I, III, etc.

The results of this work as shown in the tables seem to prove conclusively that a complete absorption is not secured in 5 minutes. The average non-tannin figures for 5 minute shaking are without exception higher than those for 10 minutes. The difference is not very great, being largest for the hemlock extract, sample C, and least for the weak liquor, sample D, where the difference is almost within the limits of experimental error. The results by the 5 minute shake are moreover with few exceptions distinctly less in agreement than those by the 10 minute shake and this was to be expected. The maximum absorption is not reached in 5 minutes and consequently the time of contact becomes a modifying factor in the non-tannin figure. As the time of contact of liquor and hide powder over and above the 5 minutes of shaking naturally varied with the different operators this variation affected the results reported. That the absorption was not complete in five minutes is further shown by the fact that a number of the 5 minute non-tannins gave a test for tannin with gelatine-salt.

It is undoubtedly possible by leaving the liquor and hide powder in contact several minutes and then shaking 5 minutes to obtain a maximum absorption and the same result as by a ten minute shaking. For instance the Referee tested this on the Official Samples allowing the liquor and hide powder to remain

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in contact 25 minutes then shaking 5 minutes with results as follows:

SAMPLE	A.	B.	C.	D.IV.	D.VI.	E.IV.	E.VI.
Shaken 10 M.....	10.97	10.77	14.34	5.69	5.51	7.18	6.67
Macerated 25 M. Shaken 5 M.	10.91	10.75	14.11	5.67	5.49	7.09	6.62

In routine work, therefore, where one had several analyses to make, and hide powder and liquor remained in contact several minutes both before and after shaking, a 5 minute shaking might suffice, but supposing a strict adherence to a 5 minute shaking with no extra time of contact beyond that necessary to put the mixture on and off the shaker and a very sensible variation of result would ensue. In the experience of the Referee, extra time of contact is immaterial when the time of shaking is ten minutes. For the above mentioned reasons and the results shown by the general work the Referee is firmly convinced that it is unwise to cut down the present time of shaking.

As regards the relative merits of the Official and the Experimental methods of liquor analysis the report of the liquor committee is in general favorable to the Official Method. To secure more experimental data the Referee called for analyses of two liquors by the two methods in the general work. The directions sent out were as follows:

Study IV. Analyze liquors D and E by the Official Method (see Journal Vol. 1, No. 3, pages 167-168.)

D contains about 6.5% solids, 1.0% tannin.
E " " 11.7% " 4.8% "

Study IVa. Determine Non-tans as by the Official Method except shake for 5 minutes.

Study V. Determine Non-tans as by the Official Method except use hide powder chromed as in study III under Extract Analysis.

Study Va. Determine Non-tans as in V except shake 5 minutes.

Study VI. Make up solutions for analysis so as to contain .35 to .45 gms. tannin per 100 cc. making dilution with water at *room* temperature. Determine Total Solids by evaporating and drying such an amount of solution as will give a residue of from .7–.8 gms.

Determine Soluble Solids as in study II under Extract Analysis except evaporate and dry the same number off cc. as used in the Total Solids determination above.

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Determine Non-tans by the Official Method for extracts, except evaporate and dry the same number of cc. as used in the Total and Soluble Solids determinations above.

Study VIa. Determine Non-tans as in VI except shake 5 minutes.

Study VII. Determine Non-tans as in VI except use hide powder chromed as in study III under extract Analysis.

Study VIIa. Determine Non-tans as in VII except shake for 5 minutes.

Before proceeding to a consideration of the results of this work the Referee would say that he found the practice of reporting results on liquors varied among the collaborators, several collaborators reporting results in percentages of volume and the rest in percentages of weight. With such a liquor as sample E this causes rather a wide variation of analysis figures. In order to avoid this variation the Referee recalculated all reports where necessary on to a weight basis and it is these values which appear in the tables. It would seem desirable that this source of variation should be done away with and the Referee recommends that an article be inserted in the method of liquor analysis to the effect that all results are to be reported in percentages of weight of liquor analyzed.

The averages of the reports by the two methods on the two liquor samples sent out, as shown in the tables, are once more not decisive as to the superiority of the one method over the other. The Official Method shows better concordance on E, the Experimental on D. The Official Method shows a lower percentage of tannin throughout. There seems little doubt, however, that this percentage is too low. From the reports of the men who tested the Non-tannin filtrates with gelatine-salt solution, in most of the filtrates where the Official Method was used, tannin was found present, this too notwithstanding that practically all the collaborators used more hide powder than called for by the method. The evidence is practically conclusive in the case of E somewhat less so in the case of D, that the amounts of hide powder called for by the Official Method are insufficient to fully detannize the solution. It is to be regretted that more of the collaborators did not make careful tests of the Non-tannin filtrates with gelatine-salt solution as directed by the Referee. The Non-tannin values reported by the experimental method are probably

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somewhat higher than the method would normally yield in consequence of the rather high value for tannin sent out by the Referee with the directions and the consequently weaker dilution used for analysis. From a careful consideration of all the data it seems probable that the actual percentage of tannin in the two liquors lies between the values obtained by the two methods, that is, if we may base our conclusions on the non-tannin filtrate of greatest weight which yet gives no test with gelatine-salt. The question is complicated by that of selective absorption, some results seeming to indicate that when two hide powders are so used as to give the largest non-tannin residue that gives no test with gelatine-salt the two residues so obtained may vary considerably, due to a larger absorption of non-tannins by one hide than the other. The question of what is or what is not the correct percentage of tannin to report for a liquor seems therefore distinctly complicated and neither the Committee results nor the results from the collaborative work seem to show the way to a definite decision. The Referee while of the opinion that a method based on the present experimental method is preferable and calculated to yield figures more in harmony with the analytical figures obtained by the present method for other tanning materials deems the evidence so far obtained insufficient to warrant him in recommending its adoption in place of the present Official Method. He regards this year's work as decisive in showing that the amounts of dry hide powder called for in the Official Method are insufficient to detannize the analysis solutions and recommends that a Committee be appointed to revise these amounts at as early a date as possible.

To summarize, the Referee regards it as desirable that work be done on the following subjects:

A method for the analysis of leather and the determination of nitrogen.

A method for color tests with skivers.

A method for the qualitative and quantitative determination of the different tanning materials.

The value of tests with mordanted cotton.

Methods for the analysis of tannery acids.

A method for the determination of free acids in liquors.

Effect of various methods of chroming and washing hide powder.

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Solubility of the chromed hide powder or any of its constituents in the analysis solution.

Effect of varying acidity of hide powder on the analysis figures.

Effect of acid in liquors on the analysis figures.

Determination of the tannin value of a liquor most in harmony with the values obtained in the analysis of fresh tanning materials, and a method by which this value may be obtained.

The Referee recommends that First: The present method of Soluble Solids determination be replaced by the following: To 1 gm. of Kaolin in a beaker add 75 c. c. of solution; stir and pour on 590 S. and S. 15 c. c. pleated filtered paper; return filtrate to paper for one hour keeping filter full. At end of hour pour solution from filter or remove with pipette. Bring 800 c. c. of solution to 20° C; refill the filter with this solution and begin to collect filtrate for evaporating and drying so soon as filtrate becomes *Clear*, keep filter full.

Second, that an alternative filter paper be adopted for the Soluble Solids determination and he suggests Dreverhoff No. 400.

Third, that a method be drawn up for determining the acidity of hide powder, and that limiting values be assigned within which an official hide powder must fall.

Fourth, that provision be made to secure uniformity in reporting results of liquor analyses.

Fifth, that the amounts of hide powder called for by the Official Method for liquor analysis be revised.

It only remains for the Referee to express his thanks to all those who have assisted him in this work, the Chairman of Committees and especially his Assistant Referee, Mr. Wilson who has aided him with much valuable advice.

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ABBREVIATIONS USED IN TABLES.

I.—Study I Official method of extract analysis.

II.—Study II Provisional method of soluble solids determination.

III.—Study III Official method for non-tannin determination, except using heavily chromed hide powder.

IV.—Study IV Official method of liquor analysis.

V.—Study V Official method of liquor analysis for non-tannin, except using heavily chromed hide powder.

VI.—Study VI Provisional method of liquor analysis.

VII.—Study VII Provisional method of liquor analysis for non-tannins, except using a heavily chromed hide powder.

Ia.—VIIa. Same as studies I to VII respectively for non-tannins, except shake five minutes.

T. S.—Total Solids.

S. S.—Soluble Solids.

Time—Time in minutes to collect Soluble Solids for evaporating and drying.

Filt.—Character of filtrate.

B. or Bl., Brilliant; C. or Cl., Clear; O. or Op., Opalescent; Cd., Cloudy; N., no test for tannin with gelatine-salt solution.

T.—gave test for tannin with gelatine-salt solution.

Temp.—Room temperature in degrees centigrade during operation.

N. T.—Non-Tannin.

Ins.—Insolubles.

Acid in H. P.—Number of cc. of tenth normal alkali to neutralize 5 gms. of the hide powder used for analysis.

Dry H. P.—Number of grams of dry hide powder used per 200 cc. of analysis solution.

Moist.—Percentage of moisture in wet hide powder used.

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SAMPLE A.

	T. S.	I S. S.	Time Minutes	Filt.	Temp.	II S. S.	Time	Filt.	Temp.	I Ins.	II Ins.
Ackermann	88.50	80.67	418	Op.	22	79.92	177	Cl.	22	7.83	8.58
Alden and Stauffer	86.46	78.00	285	Bl.	22	77.97	180	Bl.	22	8.46	8.49
Cushing and Maxwell	88.70	79.94	405	Op.	22	80.00	270	Op.	22	8.76	8.70
Eachus	87.29	81.10	...	...	..	80.58	...	...	..	6.19	6.71
Evans	87.40	80.30	190	Bl.	23	79.58	120	Bl.	23	7.10	7.82
Fox	88.13	80.25	255	Cl.	23	80.17	180	Cl.	23	7.88	7.96
Hoppenstedt	87.58	79.42	287	Cl.	21	79.46	176	Op.	21	8.16	8.12
Loveland	88.32	80.60	145	Cl.	24	79.65	128	Bl.	24	7.72	8.67
Mosbaugh*	88.58	82.57	...	...	..	82.27	...	...	..	6.01	6.31
Mosser	86.41	79.58	285	Cl.	22	79.42	205	Cl.	22	6.83	6.99
Nichols	88.00	78.67	...	Cl.	21	79.67	...	Cl.	21	9.33	8.33
Norris	86.98	79.22	320	Cl.	25	80.24	146	Cl.	24	7.76	6.74
Reed	87.27	79.01	260	Cl.	21	78.77	161	Bl.	21	8.26	8.50
Small	86 99	79.27	314	Cl.	25	79.24	240	Cl.	25	7.72	7.75
Teas	88.24	80.92	160	Bl.	22	80.59	110	Bl.	22	7.32	7.65
Veitch	87.59	79.83	205	Cl.	24	79.67	170	Cl.	24	7.76	7.92
White	87.36	78.85	210	Cl.	23	78.08	214	Cl.	23	8.51	9.28
Wilson	88.13	80.54	145	Cl.	23	79.63	128	Bl.	23	7.59	8.50
Worthen	87.55	79.17	348	Op.	24	79.60	165	Op.	24	8.38	7.95
Average	87.61	79.74	265			79.57	173			7.87	8.04
Maximum	88.70	81.10	418			80.59	270			9.33	9.28
Minimum	86.41	78.00	145			77.97	110			6.19	6.71
Greatest Diff.	1.20	1.74				1.60				1.68	1.33

* Omitted from average.

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SAMPLE B.

	T.S.	I. S.S.	Time	Filt.	Temp.	II. S.S.	Time	Filt.	Temp.	I. Ins.	II. Ins.
Ackermann	46.51	46.18	68	Bl.	21	46.16	102	Bl.	22	.33	.35
Alden and Stauffer	45.45	45.01	75	Bl.	22	44.67	100	Bl.	22	.44	.78
Cushing and Maxwell	46.35	46.19	225	Bl.	22	46.05	150	Bl.	22	.16	.30
Eachus	45.78	45.72	..	..	..	45.70	..	..	..	.06	.08
Evans	46.40	46.40		No analysis		46.40		No analysis		.00	.00
Fox	46.32	45.88	65	Bl.	23	45.90	95	Bl.	23	.44	.42
Hoppenstedt	46.10	45.81	60	Bl.	21	45.74	100	Bl.	21	.29	.36
Loveland	46.40	45.73	57	Bl.	24	45.79	85	Bl.	24	.67	.61
Mosbaugh*	46.35	45.84	..	..	..	45.51	..	..	..	.51	.84
Mosser	45.83	45.58	55	Bl.	22	45.50	80	Bl.	22	.25	.33
Nichols	46.25	45.83	..	Cl.	22	45.67	..	Cl.	22	.42	.58
Norris	46.02	45.63	76	Cl.	25	46.02	96	Cl.	24	.39	.00
Reed	45.77	45.37	47	Bl.	21	45.40	81	Bl.	21	.40	.37
Small	45.73	45.22	59	Bl.	25	45.40	95	Bl.	24	.51	.33
Teas	46.23	46.23		No analysis.		46.23		No analysis.		.00	.00
Veitch	45.96	45.76	117	Bl.	24	45.58	105	Bl.	24	.20	.38
White	45.41	45.41	85	Cl.	22	45.40	114	Cl.	22	.00	.01
Wilson	46.69	46.03	57	Bl.	23	46.30	85	Bl.	23	.66	.39
Worthen	45.87	45.42	60	Bl.	24	45.37	87	Bl.	24	.45	.50
Average	46.06	45.74	79	..	..	45.74	98	..	..	.32	.32
Maximum	46.69	46.40	225	..	..	46.40	150	..	..	.67	.78
Minimum	45.41	45.01	47	..	..	44.67	80	..	..	.00	.01
Greatest Diff.	1.28	.73	..	..	..	1.07	..	..	..	.35	.46

* Omitted from average.

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SAMPLE C.

	T.S.	I. S.S.	Time	Filt.	Temp.	II. S.S.	Time	Filt.	Temp.	I. Ins.	II. Ins.
Ackermann	46.87	43.86	435	Op.	22	43.75	186	Op.	22	3.01	3.12
Alden and Stauffer	46.00	43.33	210	Bl.	22	42.86	170	Bl.	22	2.67	3.14
Cushing and Maxwell	46.65	43.33	315	Bl.	22	43.57	240	Bl.	22	3.32	3.08
Eachus	46.87	44.25	..	..	..	44.09	..	..	..	2.62	2.78
Evans	45.93	44.02	..	Bl.	23	43.40	100	Cl.	23	1.91	2.53
Fox	46.88	43.91	230	Cl.	23	43.91	200	Cl.	23	2.97	2.97
Hoppenstedt	46.72	43.40	288	Bl.	21	43.42	206	Bl.	21	3.32	3.30
Loveland	46.84	43.92	160	Bl.	24	43.72	155	Bl.	24	2.92	3.12
Mosbaugh*	46.81	43.80	..	..	..	43.47	..	..	..	3.01	3.34
Mosser	46.40	43.96	345	Cl.	22	43.63	205	Cl.	22	2.44	2.77
Nichols	46.71	43.43	..	Cl.	22	43.14	..	Cl.	22	3.28	3.57
Norris	46.59	43.64	307	Cl.	25	43.50	160	Cl.	24	2.95	3.09
Reed	46.66	43.63	156	Cl.	21	43.54	166	Bl.	21	3.03	3.12
Small	46.59	43.85	142	Cl.	25	43.58	180	Bl.	25	2.74	3.01
Teas	46.60	43.65	155	Bl.	22	43.51	100	Bl.	22	2.95	3.09
Veitch	46.48	43.65	238	Cl.	24	43.18	185	Cl.	24	2.83	3.30
White	46.30	43.00	210	Cl.	22	43.08	214	Cl.	22	3.30	3.22
Wilson	46.72	43.94	160	Bl.	23	43.72	155	Bl.	23	2.78	3.00
Worthen	46.68	43.74	185	Cl.	24	43.48	144	Cl.	24	2.94	3.20
Average	46.58	43.69	236	..	..	43.50	173	..	..	2.89	3.08
Maximum	46.88	44.25	435	..	..	44.09	240	..	..	3.32	3.57
Minimum	45.93	43.00	142	..	..	42.86	100	..	..	1.91	2.53
Greatest Diff.	.65	.69	..	..	..	.64	..	..	..	.98	.55

* Omitted from average.

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SAMPLE D.

	Study IV							Study VI								
	Sp. Gr.	Dil.	T. S.	S. S.	Time	Filt.	Temp.	Dil.	c. c. Evap.	T. S.	S. S.	Time	Filt.	Temp.	IV Ins.	VI Ins.
Ackerman	1.0272	105	6.54	6.29	107	Bl.	22	400	27	6.54	6.21	125	Op.	22	.25	.33
Alden and Stauffer	1.029	111	6.49	6.25	100	Cl.	23	400	30	6.51	6.29	130	Cl.	21	.24	.22
Cushing and Maxwell	1.0284	120	6.55	6.25	315	Bl.	22	350	35	6.54	6.26	180	Bl.	22	.30	.28
Evans	.....	110	6.65	6.35	...	Bl.	23	400	35	6.57	6.34	90	Bl.	23	.30	.23
Fox	1.0270	110	6.58	6.33	125	Bl.	23	400	27	6.59	6.33	125	Bl.	23	.25	.26
Hoppenstedt	1.0266	110	6.54	6.31	125	Bl.	21	400	27	6.58	6.31	110	Bl.	21	.23	.27
Loveland	1.0279	110	6.52	6.32	86	Bl.	23	400	30	6.52	6.26	90	Bl.	23	.20	.26
Mosbaugh	.....	100	6.53	6.28	..	..	..	400	30	6.55	6.27	..	...	..	.25	.28
Mosser	1.030	150	6.40	6.23	165	Bl.	22	500	22	6.55	6.32	120	Cd.	23	.17	.23
Norris	1.0284	102	6.49	6.26	110	Cl.	24	437	25	6.52	6.25	130	Cl.	23	.23	.27
Reed	1.0267	105	6.56	6.31	125	Bl.	22	380	30	6.54	6.31	113	Bl.	22	.25	.23
Small	1.0283	105	6.54	6.29	150	Bl.	24	450	25	6.57	6.32	115	Bl.	24	.25	.25
Teas	.....	115	6.57	6.36	155	Bl.	22	400	35	6.59	6.35	90	Bl.	22	.21	.24
Veitch	1.0240	100	6.43	6.17	130	Bl.	24	400	30	6.47	6.24	135	Bl.	24	.26	.23
White	1.0275	100	6.45	6.24	65	Cl.	23	400	30	6.63	6.37	80	Cl.	23	.21	.26
Wilson	1.0279	110	6.62	6.36	86	Bl.	23	400	30	6.57	6.29	90	Bl.	23	.26	.28
Worthen	1.0283	105	6.53	6.29	121	Cl.	23	440	25	6.54	6.30	122	Bl.	23	.24	.24
Average			6.53	6.29	131					6.55	6.30	115			.24	.25
Maximum			6.65	6.36	315					6.63	6.37	180			.30	.33
Minimum			6.40	6.17	65					6.47	6.21	80			.17	.22
Greatest Diff.			.13	.12						.08	.09				.07	.08

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SAMPLE E.

	STUDY IV.								STUDY VI.						
	Sp. gr.	Dil.	T.S.	S.S.	Time	Filt.	Temp,	Dil.	c. c. Evap.	T.S.	S.S.	Time	Filt.	Temp.	IV. Ins.	VI. Ins.
Ackermann	1.0489	55	11.66	11.16	86	Bl.	21	80	72	11.57	11.05	97	Op.	21	.50	.52
Alden and Stauffer	1.051	55	11.42	11.01	105	Cl.	23	83	75	11.61	11.00	130	Cl.	23	.41	.61
Cushing and Maxwell	1.0504	60	11.64	11.28	255	Cl.	22	75	75	11.62	11.17	210	Bl.	22	.36	.45
Evans	....	60	11.67	11.21	105	Bl.	23	80	75	11.87	11.29	85	Bl.	23	.46	.58
Fox	1.0485	60	11.73	11.30	99	Bl.	23	85	72	11.63	11.11	120	Cl.	23	.43	.52
Hoppenstedt	1.0480	60	11.67	11.17	93	Bl.	21	85	72	11.59	11.17	100	Bl.	21	.50	.42
Loveland	1.0495	60	11.56	11.14	65	Bl.	23	85	75	11.61	11.25	85	Bl.	23	.42	.36
Mosbaugh	....	60	11.53	11.13	..	..	..	85	75	11.58	11.12	..	..	..	.40	.46
Mosser	1.054	60	11.36	10.96	105	Bl.	22	80	85	11.58	10.90	120	Bl.	23	.40	.68
Norris	1.0503	57	11.52	11.15	91	Cl.	24	84	70	11.47	10.99	130	Cl.	23	.37	.48
Reed	1.0482	55	11.67	11.20	74	Bl.	22	80	80	11.63	11.19	105	Bl.	22	.47	.44
Small	1.0499	60	11.60	11.17	90	Bl.	23	80	75	11.59	11.13	110	Bl.	23	.43	.46
Teas	....	65	11.67	11.28	95	Bl.	22	80	75	11.73	11.21	85	Bl.	22	.39	.52
Veitch	1.045	60	11.48	11.07	125	Bl.	24	80	80	11.49	11.04	120	Bl.	24	.41	.45
White	1.050	75	11.49	11.19	80	..	..	85	80	11.51	10.99	80	Cl.	23	.30	.52
Wilson	1.0495	60	11.68	11.20	65	Bl.	23	85	75	11.72	11.24	85	Bl.	23	.48	.48
Worthen	1.0499	60	11.53	11.16	81	Bl.	24	80	75	11.53	11.10	114	Bl.	23	.37	.43
Average	....	..	11.58	11.16	101	..	..	..	..	11.61	11.12	111	..	..	.42	.49
Maximum	....	..	11.73	11.30	255	..	..	..	..	11.87	11.29	210	..	..	.50	.68
Minimum	....	..	11.36	10.96	65	..	..	..	..	11.47	10.90	80	..	..	.30	.36
Greatest Diff.	....	..	.22	.20	..	..	..	..	..	.26	.22	..	..	..	.12	.19

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SAMPLE A.

	Acid	I		Ia		Dry		III		IIIa		Dry		I	III
	H. P.	N. T.	Filt.	N. T.	Filt.	H. P.	Moist	N. T.	Filt.	N. T.	Filt.	H. P.	Moist	T. S.—N.T.	T.S.—N.T.
Ackerman	11.0	11.67	C ·	11.90	C ·	12.0	76.	12.30	C ·	12.59	C ·	12.5	75.	76.83	76.20
Alden and Stauffer	1.7	11.85	O ·	12.34	O ·	11.9	76.7	10.23	C ·	10.84	C ·	13.8	71.7	74.61	76.23
Cushing and Maxwell	14.1	12.47	C ·	12.45	C ·	11.1	77.8	12.53	B ·	13.06	B ·	11.3	77.5	76.23	76.17
Eachus	8.2	11.30	· ·	11.21	· ·	....	....	11.51	· ·	11.66	· ·	....	....	75.99	75.78
Evans	...	10.26	· ·	10.92	· ·	13.3	72.5	11.56	· ·	12.14	· ·	13.5	70.	77.14	75.84
Fox	11.0	11.35	C ·	11.65	C ·	12.5	75.	11.50	C ·	11.85	C ·	13.0	74.	76.78	76.63
Hoppenstedt	11.8	11.21	C ·	11.65	C ·	13.0	74.0	11.46	C ·	11.75	C ·	13.0	74.0	76.37	76.12
Loveland	3.7	11.13	B N	11.48	B N	13.6	69.8	11.35	B N	11.68	B N	14.8	69.2	77.19	76.97
Mosbaugh*	4.2	14.88	· ·	15.19	· ·	....	....	14.89	· ·	15.01	· ·	....	....	73.70	73.69
Mosser	10.0	12.09	B ·	12.00	B ·	12.5	75.	12.17	B ·	11.70	B ·	10.4	74.	74.32	74.24
Nichols	13.9	11.30	C ·	11.89	C ·	11.3	75.	12.70	C ·	12.12	C ·	9.2	77.	76.70	75.30
Norris	9.2	10.23	C N	10.31	C N	13.7	72.9	10.67	C N	10.86	C N	15.4	69.3	76.75	76.31
Reed	10.0	10.75	B N	11.14	C N	12.4	75.0	12.74	B N	12.68	B N	12.2	74.0	76.52	74.53
Small	10.7	10.91	B N	11.81	B T	12.6	72.7	10.76	B N	11.50	B N	13.1	70.9	76.08	76.23
Teas	5.5	11.20	B ·	11.59	B ·	12.6	72.	11.56	B ·	12.14	B ·	13.5	70.	77.04	76.68
Veitch	.4	12.38	C T	13.32	C T	11.4	77.2	10.83	C N	11.32	C N	11.6	76.7	75.21	76.76
White	11.8	11.64	C ·	11.58	C ·	12.4	75.3	12.03	C ·	12.75	C ·	12.3	70.7	75.72	75.33
Wilson	3.7	10.94	B N	11.61	B N	13.8	71.2	10.90	B N	11.93	B N	13.4	70.3	77.19	77.23
Worthen	9.4	10.60	B N	11.52	B T	12.9	72.7	11.09	B N	11.90	B T	13.4	70.4	76.95	76.46
Average		11.29		11.68				11.54		11.91				76.31	76.06
Maximum		12.47		13.32				12.74		13.06				77.19	77.23
Minimum		10.23		10.31				10.23		10.84				74.32	74.24
Greatest Diff.		1.18		1.64				1.31		1.15				1.99	1.82

* Omitted from average.

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SAMPLE B.

	I N.T.	Filt.	Ia N.T.	Filt.	Dry H. P.	Moist	III N. T.	Filt	III N. T.	Filt	Dry H. P.	Moist	I T. S.- N. T.	III T. S.-N.T.
Ackermann	10.73	C —	11.07	O --	12.	76.	11.17	C —	11.45	C —	12.5	75.	35.78	35.34
Alden and Stauffer	10.43	C —	10.66	C —	11.9	76.7	10.15	C —	10.11	C —	13.8	71.7	35.02	35.30
Cushing and Maxwell	10.95	B —	11.02	B —	11.1	77.8	10.91	B —	11.15	B —	11.3	77.5	35.40	35.44
Eachus	10.70	— —	10.61	— —	—	—	10.62	— —	10.64	— —	—	—	35.08	35.16
Evans	11.18	B —	11.23	B —	12.6	72.	11.23	B —	11.52	B —	13.5	70.	35.22	35.17
Fox	10.79	B —	10.77	B —	12.5	75.	10.94	B —	10.94	B —	13.0	74.	35.53	35.38
Hoppenstedt	10.65	C —	10.85	C —	13.	74.	10.90	C —	10.88	C —	13.	74.	35.45	35.20
Loveland	12.42	B N	12.36	O T	13.6	69.8	11.75	B N	11.94	B N	10.8	69.2	33.98	34.65
Mosbaugh*	11.80	— —	11.98	— —	—	—	12.09	— —	12.02	— —	—	—	34.55	34.26
Mosser	11.03	B —	11.25	B —	12.5	75.	10.83	B —	10.78	B —	10.4	74.	34.80	35.00
Nichols	9.91	C —	10.82	— —	11.3	75.	11.12	C —	10.45	C —	9.2	77.	36.34	35.13
Norris	10.48	C N	10.52	C N	13.6	72.9	10.63	C N	10.68	C N	15.4	69.3	35.54	35.39
Reed	10.51	B N	10.92	O N	12.4	75.2	11.11	B N	11.36	B N	12.2	74.0	35.26	34.66
Small	10.75	B N	11.14	C N	12.6	72.7	10.55	B N	10.90	B N	13.1	70.9	34.98	35.18
Teas	11.05	B —	11.24	B —	12.6	72.	10.93	B —	11.60	B —	13.5	70.	35.18	35.30
Veitch	11.48	O T	11.53	O T	11.4	77.2	10.93	C N	10.97	C N	11.6	76.8	34.48	35.03
White	10.40	C —	10.81	C —	12.4	75.3	10.76	C —	11.31	C —	12.3	70.7	35.01	34.65
Wilson	12.38	B N	12.83	C T	13.8	71.2	11.63	B N	11.87	B N	13.4	70.3	34.31	35.06
Worthen	10.67	B N	10.78	C T	12.9	72.7	10.84	B N	11.07	C T	13.4	70.4	35.20	35.03
Average	10.92	....	11.13	....	....	....	10.94	....	11.09	....	....	....	35.14	35.12
Maximum	12.42	....	12.83	....	....	....	11.75	....	11.94	....	....	....	36.34	35.44
Minimum	9.91	....	10.52	....	....	....	10.15	....	10.11	....	....	....	33.98	34.65
Greatest Diff.	1.50	....	1.70	....	....	....	.81	....	.98	....	....	....	1.20	.79

*Omitted from average

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SAMPLE C.

	I. N.T.	Filt.	Ia. N.T.	Filt.	Dry H.P.	Moist.	III. N.T.	Filt.	IIIa. N.T.	Filt.	Dry H.P.	Moist.	I. T.S.-N.T.	III. T.S.-N.T.
Ackermann	13.91	C —	14.95	O —	12.	76.	14.34	C —	14.53	C —	12.5	75.	32.96	32.53
Alden and Stauffer	13.36	O —	14.04	O —	11.9	76.7	13.10	C —	13.02	C —	13.8	71.7	32.64	32.90
Cushing and Maxwell	14.39	B —	14.97	B —	11.1	77.8	14.45	B —	14.67	B —	11.3	77.5	32.26	32.20
Eachus	14.01	— —	13.70	— —	..	..	14.11	— —	13.76	— —	..	..	32.86	32.76
Evans	13.71	C —	13.92	C —	13.3	72.5	13.94	C —	14.29	C —	12.6	70.	32.22	31.99
Fox	13.86	C —	13.88	Cd —	12.5	75.	13.90	C —	14.03	C —	13.0	74.	33.02	32.98
Hoppenstedt	14.04	O —	14.38	O —	13.0	74.	14.00	C —	14.13	C —	13.0	74.0	32.68	32.72
Loveland	13.92	C N	14.26	O T	13.6	69.8	14.09	C N	14.36	O T	14.8	69.2	32.92	32.75
Mosbaugh *	15.40	— —	15.73	— —	..	..	15.50	— —	15.96	— —	..	..	31.41	31.31
Mosser	14.24	C —	14.24	C —	12.5	75.	14.25	C —	14.62	O —	10.4	74.	32.16	32.15
Nichols	13.85	C —	13.44	C —	11.3	75.	14.10	C —	14.68	C —	9.20	77.	32.86	32.61
Norris	13.50	C N	13.73	C N	13.6	72.9	13.55	C N	13.63	C N	15.4	69.3	33.09	33.04
Reed	13.75	O N	15.64	Cd N	12.4	75.2	14.98	C N	15.95	O N	12.2	74.0	32.91	31.68
Small	14.11	B N	15.52	O T	12.6	72.7	13.89	B N	14.58	O T	13.1	70.9	32.48	32.70
Teas	14.16	B —	14.29	B —	12.6	72.	13.94	B —	14.18	B —	13.5	70.	32.44	32.66
Veitch	14.83	O T	15.38	O T	11.4	77.2	14.47	O T	14.60	O T	11.6	76.8	31.65	32.01
White	13.64	C —	14.24	C —	12.4	75.3	14.99	O —	16.40	O —	12.3	70.7	32.66	31.31
Wilson	14.07	C N	14.23	O T	13.8	71.2	13.82	C N	14.48	O T	13.4	70.3	32.65	32.90
Worthen	13.74	C N	14.64	O T	12.9	72.7	13.78	C N	14.76	O T	13.4	70.4	32.94	32.90
Average	13.95	..	14.41	....	....	....	14.09	....	14.48	....	....	....	32.63	32.49
Maximum	14.83	....	15.64	....	....	....	14.99	....	16.40	....	....	....	33.09	33.04
Minimum	13.36	....	13.44	....	....	....	13.10	....	13.03	....	....	....	31.65	31.31
Greatest Diff.	.88	....	1.23	....	....	....	.99	....	1.92	....	....	....	.98	1.18

* Omitted from average.

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SAMPLE D.

	IV. N.T.	Filt.	IVa. N.T.	Filt.	Dry H.P.	Moist.	V. N.T.	Filt.	Va. N.T.	Filt.	Dry H.P.	Moist.	IV. T.S.–N.T.	V. T.S.–N.T.
Ackermann	5.54	C —	5.60	C —	1.92	76.	5.52	C —	5.58	O —	2.00	75.	1.00	1.02
Alden and Stauffer	5.51	C —	5.58	C —	2.04	77.3	5.56	C —	5.60	C —	2.6	71.2	.98	.93
Cushing and Maxwell	5.68	B —	5.77	B —	1.6	77.8	5.65	B —	5.72	B —	1.6	77.5	.87	.90
Evans	5.61	C —	5.68	C —	2.24	72.	5.61	C —	5.65	C —	2.4	70.	1.04	1.04
Fox	5.58	C —	5.62	C —	2.00	75.	5.53	C —	5.59	C —	2.08	74.	1.00	1.05
Hoppenstedt	5.61	C —	5.61	C —	2.08	74.	5.53	C —	5.57	C —	2.08	74.	.93	1.01
Loveland	5.55	C N	5.60	C N	2.19	68.7	5.56	C N	5.55	C T	2.16	69.2	.97	.96
Mosbaugh	5.43	— —	5.52	— —	2.2	..	5.45	— —	5.54	— —	2.3	..	1.10	1.08
Mosser	5.53	C —	5.54	C —	2.5	75.	5.56	C —	5.61	C —	2.6	74.	.87	.84
Norris	5.44	C N	5.46	C T	2.61	73.9	5.35	C N	5.39	C T	2.96	70.3	1.05	1.14
Reed	5.73	C N	5.79	C N	1.52	75.2	5.69	C N	5.75	C N	1.56	73.0	.83	.87
Small	5.67	B T	5.74	B T	1.84	73.7	5.60	B T	5.62	B T	2.01	71.3	.87	.94
Teas	5.52	C —	5.63	Cd —	2.24	72.	5.54	B —	5.56	C —	2.4	70.	1.05	1.03
Veitch	5.46	B —	5.48	B —	1.8	77.2	5.40	B —	..	..	1.9	76.7	.97	1.03
White	5.56	C —	5.57	C —	1.7	79.0	5.50	C —	5.62	C —	2.1	70.5	.89	.95
Wilson	5.54	C N	5.61	C N	2.06	70.6	5.56	C N	5.64	C T	1.97	71.9	1.08	1.06
Worthen	5.72	B T	5.75	B T	1.37	72.5	5.70	B T	5.72	C T	1.37	71.5	.81	.83
Average	5.57	....	5.62	....	..	..	5.54	....	5.57	....	..	..	.96	.99
Maximum	5.73	....	5.79	....	..	..	5.70	....	5.75	....	..	..	1.10	1.14
Minimum	5.43	....	5.46	....	..	..	5.35	....	5.39	....	..	..	.81	.83
Greatest Diff.	.16	....	.17	....	..	..	.19	....	.18	....	..	..	.15	.16

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SAMPLE D.

	VI N. T.	Filt.	VIa N. T.	Filt.	Dry H.P.	Moist	VII N. T.	Filt.	VIIa N. T.	Filt.	Dry H.P.	Moist	VI T.S.-N.T.	VII T.S.-N.T
Ackermann	5.37	C —	5.37	C —	12.0	76.	5.37	C —	5.40	Cd —	12.5	75.	1.17	1.17
Alden and Stauffer	5.54	C —	5.45	C —	11.2	77.3	5.35	C —	5.34	C —	12.8	71.2	.97	1.16
Cushing and Maxwell	5.52	B —	5.51	B —	11.1	77.8	5.36	B —	5.43	B —	11.3	77.5	1.02	1.18
Evans	5.47	C —	5.38	C —	11.2	72.	5.51	C —	5.46	C —	12.0	70.	1.10	1.06
Fox	5.36	C —	—	— —	12.5	75.	5.29	C —	5.36	C —	13.0	74.	1.23	1.30
Hoppenstedt	5.41	C —	5.42	C —	13.0	74.0	5.32	C —	5.36	C —	13.0	74.	1.17	1.26
Loveland	5.26	O N	5.34	O N	14.1	68.7	5.31	O N	5.33	O N	13.9	69.2	1.26	1.21
Mosbaugh	5.32	— —	5.34	— —	11.7	—	—	— —	—	— —	- -	—	1.23	- -
Mosser	5.57	Cd —	5.54	C —	8.4	76.	5.52	C —	5.55	C —	9.1	74.	.98	1.03
Norris	5.38	C N	5.38	C N	13.1	73.8	5.30	C N	5.33	C N	13.9	72.2	1.14	1.22
Reed	5.39	O N	5.46	O N	12.7	75.2	5.42	Cd N	5.50	Cd N	13.0	73.0	1.15	1.12
Small	5.49	B N	5.56	B T	12.1	73.7	5.40	B N	5.50	B N	12.6	71.3	1.08	1.17
Teas	5.53	C —	5.66	C —	11.2	72.	5.40	Cd —	5.46	C —	12.0	70.	1.06	1.19
Veitch	5.36	B —	5.35	B —	11.4	77.2	5.20	B —	5.34	B —	9.3	76.7	1.11	1.27
White	5.50	O —	5.58	O —	8.4	79.0	5.45	O —	5.43	O —	10.3	70.5	1.13	1.18
Wilson	5.30	O N	5.32	O N	13.2	70.6	5.31	O N	5.39	O N	12.7	71.8	1.27	1.26
Worthen	5.38	B T	5.44	B T	12.9	72.5	5.36	B T	5.40	C T	13.0	71.5	1.16	1.18
Average	5.42	....	5.44	....	....	....	5.37	....	5.41	....	....	....	1.13	1.18
Maximum	5.57	....	5.66	....	....	....	5.52	....	5.55	....	....	....	1.27	1.30
Minimum	5.26	....	5.32	....	....	....	5.20	....	5.33	....	....	....	.97	1.03
Greatest Diff	.16	....	.22	....	....	....	.17	....	.14	....	....	....	.16	.15

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SAMPLE D.

	IV. N.T.	Filt.	IVa. N.T.	Filt.	Dry H.P.	Moist.	V. N.T.	Filt.	Va. N.T.	Filt.	Dry H.P.	Moist.	IV. T.S.-N.T.	V. T.S.-N.T.
Ackermann	5.54	C —	5.60	C —	1.92	76.	5.52	C —	5.58	O —	2.00	75.	1.00	1.02
Alden and Stauffer	5.51	C —	5.58	C —	2.04	77.3	5.56	C —	5.60	C —	2.6	71.2	.98	.93
Cushing and Maxwell	5.68	B —	5.77	B —	1.6	77.8	5.65	B —	5.72	B —	1.6	77.5	.87	.90
Evans	5.61	C —	5.68	C —	2.24	72.	5.61	C —	5.65	C —	2.4	70.	1.04	1.04
Fox	5.58	C —	5.62	C —	2.00	75.	5.53	C —	5.59	C —	2.08	74.	1.00	1.05
Hoppenstedt	5.61	C —	5.61	C —	2.08	74.	5.53	C —	5.57	C —	2.08	74.	.93	1.01
Loveland	5.55	C N	5.60	C N	2.19	68.7	5.56	C N	5.55	C T	2.16	69.2	.97	.96
Mosbaugh	5.43	— —	5.52	— —	2.2	..	5.45	— —	5.54	— —	2.3	..	1.10	1.08
Mosser	5.53	C —	5.54	C —	2.5	75.	5.56	C —	5.61	C —	2.6	74.	.87	.84
Norris	5.44	C N	5.46	C T	2.61	73.9	5.35	C N	5.39	C T	2.96	70.3	1.05	1.14
Reed	5.73	C N	5.79	C N	1.52	75.2	5.69	C N	5.75	C N	1.56	73.0	.83	.87
Small	5.67	B T	5.74	B T	1.84	73.7	5.60	B T	5.62	B T	2.01	71.3	.87	.94
Teas	5.52	C —	5.63	Cd —	2.24	72.	5.54	B —	5.56	C —	2.4	70.	1.05	1.03
Veitch	5.46	B —	5.48	B —	1.8	77.2	5.40	B —	..	..	1.9	76.7	.97	1.03
White	5.56	C —	5.57	C —	1.7	79.0	5.50	C —	5.62	C —	2.1	70.5	.89	.95
Wilson	5.54	C N	5.61	C N	2.06	70.6	5.56	C N	5.64	C T	1.97	71.9	1.08	1.06
Worthen	5.72	B T	5.75	B T	1.37	72.5	5.70	B T	5.72	C T	1.37	71.5	.81	.83
Average	5.57	....	5.62	....	..	..	5.54	....	5.57	....	..	..	.96	.99
Maximum	5.73	....	5.79	....	..	..	5.70	....	5.75	....	..	..	1.10	1.14
Minimum	5.43	....	5.46	....	..	..	5.35	....	5.39	....	..	..	.81	.83
Greatest Diff.	.16	....	.17	....	..	..	.19	....	.18	....	..	..	.15	.16

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SAMPLE D.

	VI N. T.	Filt.	VIa N. T.	Filt.	Dry H.P.	Moist	VII N. T.	Filt.	VIIa N. T.	Filt.	Dry H.P.	Moist	VI T.S.-N.T.	VII T.S.-N.T
Ackermann	5.37	C —	5.37	C —	12.0	76.	5.37	C —	5.40	Cd —	12.5	75.	1.17	1.17
Alden and Stauffer	5.54	C —	5.45	C —	11.2	77.3	5.35	C —	5.34	C —	12.8	71.2	.97	1.16
Cushing and Maxwell	5.52	B —	5.51	B —	11.1	77.8	5.36	B —	5.43	B —	11.3	77.5	1.02	1.18
Evans	5.47	C —	5.38	C —	11.2	72.	5.51	C —	5.46	C —	12.0	70.	1.10	1.06
Fox	5.36	C —	—	— —	12.5	75.	5.29	C —	5.36	C —	13.0	74.	1.23	1.30
Hoppenstedt	5.41	C —	5.42	C —	13.0	74.0	5.32	C —	5.36	C —	13.0	74.	1.17	1.26
Loveland	5.26	O N	5.34	O N	14.1	68.7	5.31	O N	5.33	O N	13.9	69.2	1.26	1.21
Mosbaugh	5.32	— —	5.34	— —	11.7	—	—	— —	—	— —	— —	—	1.23	— —
Mosser	5.57	Cd —	5.54	C —	8.4	76.	5.52	C —	5.55	C —	9.1	74.	.98	1.03
Norris	5.38	C N	5.38	C N	13.1	73.8	5.30	C N	5.33	C N	13.9	72.2	1.14	1.22
Reed	5.39	O N	5.46	O N	12.7	75.2	5.42	Cd N	5.50	Cd N	13.0	73.0	1.15	1.12
Small	5.49	B N	5.56	B T	12.1	73.7	5.40	B N	5.50	B N	12.6	71.3	1.08	1.17
Teas	5.53	C —	5.66	C —	11.2	72.	5.40	Cd —	5.46	C —	12.0	70.	1.06	1.19
Veitch	5.36	B —	5.35	B —	11.4	77.2	5.20	B —	5.34	B —	9.3	76.7	1.11	1.27
White	5.50	O —	5.58	O —	8.4	79.0	5.45	O —	5.43	O —	10.3	70.5	1.13	1.18
Wilson	5.30	O N	5.32	O N	13.2	70.6	5.31	O N	5.39	O N	12.7	71.8	1.27	1.26
Worthen	5.38	B T	5.44	B T	12.9	72.5	5.36	B T	5.40	C T	13.0	71.5	1.16	1.18
Average	5.42	....	5.44	....	....	....	5.37	....	5.41	....	....	....	1.13	1.18
Maximum	5.57	....	5.66	....	....	....	5.52	....	5.55	....	....	....	1.27	1.30
Minimum	5.26	....	5.32	....	....	....	5.20	....	5.33	....	....	....	.97	1.03
Greatest Diff	.16	....	.22	....	....	....	.17	....	.14	....	....	....	.16	.15

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SAMPLE E.

	IV N. T.	Filt.	IVa N. T.	Filt.	Dry H. P.	Moist	V N. T.	Filt.	Va N. T.	Filt.	Dry H. P.	Moist	IV T.S.-N.T.	V T.S.-N.T
Ackerman	6.96	C —	7.10	C —	6.96	76.	7.08	C —	7.19	C —	7.25	75.	4.70	4.58
Alden and Stauffer	7.30	C —	7.24	C —	4.08	77.3	6.94	C —	7.27	C —	5.2	71.2	4.12	4.48
Cushing and Maxwell	7.28	B —	7.37	B —	5.8	77.8	7.16	B —	7.23	B —	5.85	77.5	4.36	4.48
Evans	6.81	C —	6.87	C —	8.4	72.	6.75	C —	6.75	C —	9.0	70.	4.86	4.92
Fox	6.94	C —	7.01	C —	7.0	75.	6.82	C —	6.96	C —	7.28	74.	4.79	4.91
Hoppenstedt	6.89	C —	6.92	C —	7.54	74.	6.80	C —	6.95	C —	7.54	74.	4.78	4.87
Loveland	6.86	O T	7.04	O T	6.89	68.7	6.89	C T	7.02	C T	6.78	69.2	4.70	4.67
Mosbaugh	7.02	— —	7.06	— —	7.4	— —	7.01	— —	6.95	— —	7.2	— —	4.51	4.52
Mosser	6.74	C —	6.73	B —	6.25	75.	6.93	B —	6.91	B —	6.50	74.	4.62	4.43
Norris	6.75	C N	6.84	C T	7.84	73.9	6.63	C N	6.72	C T	8.90	70.3	4.77	4.89
Reed	7.33	O N	7.44	C N	6.08	75.2	7.31	O N	7.49	C N	6.22	73.0	4.34	4.36
Small	7.09	B T	7.44	B T	6.51	73.7	7.09	B T	7.29	B T	6.60	71.3	4.51	4.51
Teas	6.83	C —	6.88	C —	8.4	72.	6.76	B —	6.76	C —	9.0	70.	4.84	4.91
Veitch	6.87	B —	7.07	B —	5.7	77.2	6.73	B —	—	— —	5.8	76.7	4.61	4.75
White	6.85	C —	6.95	C —	8.4	79.0	6.83	O —	7.05	O —	10.3	70.5	4.64	4.66
Wilson	6.94	O T	7.18	O T	6.47	70.6	7.11	C T	7.10	C T	6.18	71.9	4.74	4.57
Worthen	7.02	B T	7.18	B T	6.26	72.5	6.95	C T	7.22	C T	6.41	71.5	4.51	4.58
Average	6.97	....	7.08	....	....	....	6.93	....	7.05	....	....	....	4.61	4.65
Maximum	7.33	....	7.44	....	....	....	7.31	....	7.49	....	....	....	4.86	4.92
Minimum	6.74	....	6.73	....	....	....	6.63	....	6.72	....	....	....	4.12	4.36
Greatest Diff.	.36	....	.36	....	....	....	.38	...	.44	....	....	....	.49	.29

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SAMPLE E.

	VI. N.T.	Filt.	VIa. N.T.	Filt.	Dry H.P.	Moist.	VII. N.T.	Filt.	VIIa. N.T.	Filt.	Dry H.P.	Moist.	VI, T.S.-N.T	VII. T.S.-N.T.
Ackermann	6.44	C —	6.69	C —	12.0	76.	6.59	C —	6.61	C —	12.5	75.	5.13	4.98
Alden and Stauffer	6.97	C —	7.07	C —	11.2	77.3	7.08	C —	7.13	C —	12.8	71.2	4.64	4.53
Cushing and Maxwell	6.85	B —	6.94	B —	11.1	77.8	6.72	B —	6.80	B —	11.3	77.5	4.77	4.90
Evans	6.70	B —	6.71	B —	10.6	73.5	6.57	B —	6.81	B —	12.0	70.	5.17	5.30
Fox	6.48	C —	6.54	C —	12.5	75.	6.46	C —	6.58	C —	13.0	74.	5.15	5.17
Hoppenstedt	6.52	C —	6.55	C —	13.0	74.	6.39	C —	6.50	C —	13.0	74.	5.07	5.20
Loveland	6.51	O N	6.55	O N	14.1	68.7	6.55	O N	6.60	O N	13.9	69.2	5.10	5.06
Mosbaugh	6.90	....	6.99	....	11.7	..	..	....	..	....	..	..	4.68	..
Mosser	7.08	C —	7.05	C —	8.4	76.	7.05	C —	7.05	C —	9.1	74.	4.50	4.53
Norris	6.51	C N	6.54	C N	13.1	73.8	6.46	C N	6.49	C N	13.9	72.2	4.96	5.01
Reed	6.74	O N	6.86	O N	12.7	75.2	6.94	O N	7.04	O N	13.0	73.0	4.89	4.69
Small	6.67	B N	6.92	B T	12.1	73.7	6.55	B N	6.75	O T	12.6	71.3	4.92	5.04
Teas	6.72	B —	6.73	C —	10.6	73.45	6.60	B —	6.82	B —	12.0	70.0	5.01	5.13
Veitch	6.55	B —	6.57	B —	11.4	77.2	6.63	B —	6.72	B —	9.3	76.7	4.94	4.86
White	7.17	C —	7.44	C —	8.4	79.0	7.41	C —	7.38	C —	10.3	70.5	4.34	4.10
Wilson	6.51	O N	6.69	O N	13.2	70.6	6.50	O N	6.64	O N	12.7	71.8	5.21	5.22
Worthen	6.47	B N	6.62	O T	12.9	72.5	6.44	B N	6.58	C T	13.0	71.5	5.06	5.09
Average	6.68	....	6.78	....	..	..	6.69	....	6.78	....	..	..	4.93	4.92
Maximum	7.17	....	7.44	....	..	..	7.41	....	7.38	....	..	..	5.21	5.30
Minimum	6.44	....	6.54	....	..	..	6.39	....	6.49	....	..	..	4.34	4.10
Greatest Diff.	.49	....	.66	....	..	..	.72	....	.60	....	..	..	.59	.82

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